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ENSAFE

COMPREHENSIVE LONG-TERM
ENVIRONMENTAL ACTION NAVY
NAVAL BASE CHARLESTON
CHARLESTON, SOUTH CAROLINA
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FINAL
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VOLUME V
BOOK 2 of 3

AMS, INC.
MOBILE LABORATORY

AMS, INC.

MOBILE LABORATORY

QUALITY ASSURANCE PLAN

Analytical Mobile Services, Inc.
18 Timber Marsh Lane
Hilton Head, South Carolina 29926

Revision 0.0

This manual is approved in its entirety,

Christine A. LeBlanc
President

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Vice-President
Laboratory Director

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1.0 Statement of Policy

It is the policy of Analytical Mobile Services, Inc. (AMS) to maintain a Total Quality Management (TQM) program throughout the company. This philosophy dictates the implementation of standard operating procedures and quality assurance protocols for AMS' mobile analytical laboratory. This Quality Assurance (QA) manual provides a detailed explanation of work practices adhered to in the mobile laboratory to assure compliance with acceptable operating and quality assurance procedures.

The specific objectives of this QA program are as follows:

1.) Maintain adequate custody records from initial sample receipt and storage through reporting and archiving of results

2.) Use adequately trained personnel to analyze all samples by approved methods

3.) Produce defensible data with associated documentation to show each system was calibrated and operating within precision and accuracy control limits

4.) Document all the above activities in order that all data can be independently validated.

AMS' mobile laboratory intends to follow all procedures referenced in this plan and to conform to EPA and state regulatory agency guidelines for each sample analyzed. Any changes in EPA or other regulatory procedures will be incorporated during periodic revisions of this plan.

This QA plan was developed using the guidelines presented in the following manuals: "Quality Assurance Principles for Analytical Laboratories," AOAC 1991; and "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans," EPA 1983.

The purpose of this document is to assure all analyses performed by AMS' mobile laboratory are done to exacting specifications and meet all applicable QA requirements. The consistent delivery of high quality, defensible results with the appropriate QA/QC data is the ultimate goal of AMS' mobile laboratory. Strict adherence to the work practices addressed herein is the method chosen to obtain that goal.

2.0 Organizational Structure and Responsibilities

The individuals associated with the mobile laboratory are chosen based upon their relative experience, educational background and their ability to successfully meet their responsibilities within the laboratory.

2.1 Laboratory Chemist

The chemist employed to operate the mobile laboratory will possess, at a minimum, a BS degree in Chemistry or directly related field. The chemist is directly responsible for the following laboratory functions:

1.) Individual sample analyses
2.) Equipment maintenance and calibration
3.) Standard and reagent preparation
4.) Initial verification of analytical data

The chemist is also responsible for generating all applicable QA/QC data and attaching it to the raw analytical data gathered during analysis. The chemist is solely responsible for all data generated during analyses and is the person who issues the analytical reports along with all requested QA/QC documentation.

2.2 Laboratory Manager

The laboratory manager will possess, at a minimum, a BS degree in Chemistry or related field and have three or more years of direct laboratory management experience. The laboratory manager is responsible for the following job functions:

1.) Data quality
2.) Equipment and supplies procurement
3.) Client relations and marketing
4.) Scheduling of work
5.) Final review and verification of all reports

The laboratory manager is ultimately responsible for the day-to-day operations of the mobile laboratory. He/she is the primary point of contact with all clients and project contractors. He/she reports directly to the president of the company.

3.0 Sample Handling Procedures

The laboratory chemist is responsible for the receipt, login and proper storage of all samples submitted to the mobile laboratory for analysis. Samples received by the mobile laboratory must be accompanied by a completed chain of custody form. Accepted samples will immediately be logged in using the chain of custody and then be stored in the refrigerator until ready for analysis.

3.1 Chain of Custody Procedures

Every set of samples submitted to AMS' mobile laboratory will be accompanied by a completed chain of custody form. At minimum, this form will contain the following:

1.) AMS project number
2.) Client/project name
3.) Identification of person(s) performing sampling
4.) Individual sample identification
5.) Date and time sampling occurred
6.) Individual sample type and quantity
7.) Analyses requested for each sample
8.) Signature of person relinquishing samples
9.) Date and time samples are submitted

Any additional information related to the samples should be included in the "Remarks" column on the far right side of the form. The chain of custody form contains a white top sheet with yellow and magenta carbon copies underneath. The magenta copy should be retained by the person submitting samples to the lab. A photocopy of the standard chain of custody form used by AMS' mobile laboratory is found in Appendix I.

3.3 Sample Receipt and Login

All samples received at the mobile laboratory are logged in through the chain of custody in use for the specific project. The use of unique sample numbers is not necessary in the mobile lab setting due to the absence of samples from other projects. The lab performs work exclusively for the client on-site and analyzes their samples only. This makes it impossible for samples to be mixed up with samples from other jobs. The lab chemist visually inspects each sample for any discrepancies between the information listed on the chain of custody form and the information on the sample label. During this process samples are never left unattended.

3.5 Sample Preservation, Storage and Disposal

Samples taken in the field and immediately submitted to AMS' mobile laboratory for analysis are typically not preserved other than being stored at 4° Celsius. This is due to the laboratory being instantly accessible and the analysis typically being carried out within the hour. If, however, it is apparent that samples submitted will be subjected to holding time prior to analysis, acidification of volatile organic samples should be performed. Typically, a sulfuric acid solution is used to lower the pH of a water sample to <2. This acidification is only necessary for samples taken for volatile organic analysis. Holding time for a preserved volatile organic sample is 14 days (stored at 4 C) and 7 days (stored at 4 C) for semi-volatile organic analysis.

Once samples have been successfully received by the mobile laboratory, they are either analyzed immediately or stored in the laboratory refrigerator. The refrigerator is temperature monitored by a thermometer with readings recorded twice a day. All samples are analyzed within the guidelines for holding time as recommended by EPA.

Samples are held under chain of custody procedures for thirty days at which time the samples are segregated into various waste streams grouped for disposal by a licensed waste removal firm.

4.0 General Laboratory Procedures

In order to assure all samples received by the mobile analytical laboratory are analyzed in a consistent manner, EPA-approved methods are employed for all analyses. All test results are reported at the level of accuracy and precision stated in the test methods. Various standard operating procedures are used to maintain the level of consistency required for acceptable analytical results. These may include glassware preparation, standard analysis procedures, reagent preparation, instrument calibration and instrument maintenance.

4.1 Sample Bottle Preparation

All sample containers are purchased pre-cleaned according to EPA specifications through commercial suppliers such as Eagle Picher and I-Chem. All sample containers are used only once. After use, AMS rinses the containers and sends them to an appropriate recycling center.

4.2 Analytical Glassware Preparation

All glassware used for analysis in the mobile laboratory is cleaned after each procedure according to EPA recommendations. The steps taken in the cleaning process are as follows:

1.) Rinse glassware as soon as possible after use with the last solvent utilized.
2.) Allow glassware to vent and then wash with an Alconox/water solution.
3.) Triple rinse glassware with tap water and de-ionized water.
4.) Final rinse with HPLC grade methanol.
5.) Rinse with solvent to be used immediately before beginning procedure.

4.3 Reagent Preparation

All reagents and solvents used by AMS' mobile laboratory are purchased from reputable commercial suppliers such as Fisher Scientific, Aldrich or Supelco. All solvents used in preparatory procedures are the highest purity available and meet all criteria for use in GC/MS procedures.

4.4 Analytical Standards

All standards used for internal and external calibrations are purchased from reputable commercial suppliers such as Supelco or Fisher Scientific. Internal standards, surrogates, matrix spikes, etc., are purchased as Separate Source Standards with accompanying QA/QC data packets. This process enables AMS' mobile laboratory to

meet EPA requirements for using calibration and quality control reference samples from separate or independent sources in performing environmental analysis.

4.5 Standard Analytical Procedures

All procedures carried out in AMS' mobile laboratory utilize state-of-the-art analytical equipment and follow methods found in EPA's SW-846 publication, 3rd edition. In particular, Methods 3510, 3550, 5030, 8260 and 8270 are used by AMS' mobile laboratory as guidelines for writing individual Standard Operating Procedures (SOP). Copies of AMS' SOP for volatile and semi-volatile organic analysis can be found in Appendix II.

4.6 Instrument Calibration and Maintenance

Specific calibration procedures are contained in the SW-846 methods and are followed for the applicable method. In general, initial calibration requirements are five point calibration curves with continuing calibration standards run at an intermediate concentration.

Instrument re-calibration is performed when continuing calibration checks indicate that a new variable has been introduced into the analysis or instrument drift has exceeded compensation limits. To assure a greater degree of confidence in the results, Separate Source Standards (as described in Sect. 4.4) are used exclusively.

Preventative maintenance is performed regularly and as recommended by the manufacturers. In particular, injection port consumables are changed weekly, columns are changed as needed, the MS ion source is cleaned as needed and vacuum pump oil is changed annually. Log books of routine maintenance are kept in the laboratory and updated as necessary. The major pieces of equipment found in AMS' mobile laboratory are as follows:

1.)	GC/MS No. 1:	Hewlett-Packard 5890 Series II/5972
2.)	Auto-sampler:	Hewlett-Packard 7673b
3.)	Data System:	Hewlett-Packard GC Chemstation with Enviroquant
4.)	GC/MS No. 2:	Hewlett-Packard 5890 Series II/5972
5.)	Purge & Trap:	Tekmar 3000 with Precept auto-sampler
6.)	Sonicator:	Fisher 550 Sonic Dismembrator

5.0 Analytical Quality Control

The key to a successful QA/QC program is strict adherence to the program during all phases of the project, including: sample storage, analysis, results validation and reporting. Laboratory quality control checks are part of each laboratory analysis and meet or exceed all applicable requirements.

5.1 Laboratory QC Checks

The laboratory employs control samples to assess the validity of the analytical results. Determination of the validity of sample results is based on the acceptance criteria being met by the control samples. The acceptance criteria for each type of control sample are defined in the appropriate method SOP. These acceptance criteria are per method requirements. Laboratory control samples which are processed in AMS' mobile laboratory are as follows (where applicable):

1.) **Lab Control Standards**: Blank spikes or lab control standards will be processed and analyzed per method requirements with each batch of samples.
2.) **Surrogates**: Appropriate surrogates will be added to all samples, standards and blanks.
3.) **Matrix Spikes**: Matrix spikes will be analyzed with each batch at a frequency of 5% of samples. If a method does not specify matrix spiking compounds the SW-846 matrix spiking compounds will be used. Matrix spikes containing all compounds will be analyzed quarterly to generate accuracy and precision limits.
4.) **Matrix Spike Duplicates**: Matrix spike duplicates will be analyzed with each batch or at a frequency of 5% of samples. Precision data are obtained only on the matrix spiking compounds.

5.2 Precision and Accuracy Limits

Control charts for precision and accuracy are initiated for each parameter upon method validation. Charts contain control limits (defined as $\pm$ 3 standard deviations). Control limits are updated annually for all parameters. Formulas used for calculations of precision and accuracy are as follows:

Precision: Relative percent difference is used to express precision between two replicate values. Precision data are derived from duplicate matrix spike results. The relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{(V1 - V2)}{(V1 + V2) / 2} \times 100$$

where V1 and V2 are values obtained by analyzing duplicate samples.

Accuracy: Accuracy control limits are produced from spike data. Percent recovery is used to express accuracy. The percent recovery (%R) is calculated as follows:

$$\%R = \frac{(R1 - R2)}{R3} \times 100$$

where:
- R1 = value obtained by analyzing the sample with the spike added
- R2 = value obtained by analyzing the sample
- R3 = concentration of spike added to the sample

5.3 Method Detection Limits

Method detection limits (MDL) are determined for all analyses currently performed by AMS' mobile laboratory. These limits are calculated according to the procedures set forth in SW-846.

Since MDL are based on the analyses of standards in reagent water they may not be useful in reporting data for environmental samples. Thus, practical quantitation limits (PQL) may be used for reporting a non-detected parameter. PQL are defined as the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions.

6.0 Data Reporting Quality Assurance

Quality assurance of reports within the laboratory consists of data review and report proofreading. The chemist's knowledge and experience with the requisite analysis and built-in quality control checks are a large portion of the overall quality process for AMS' mobile laboratory.

6.1 Corrective Action Measures

Any deviations from AMS' Standard Operating Procedures (SOP) must be noted and approved by the laboratory manager. If there are deviations in the QC that result in a standard sample being considered unacceptable then corrective action must be taken to assure that the same problem does not recur and the original deviation is corrected.

If, upon completion of an analysis, the quality control samples indicate the procedure fails the required quality control definitions, the analysis is defined to be out of control. The chemist and lab manager will gather all raw data and attempt to identify the cause of the problem. Once this has been determined, a discrepancy report will be issued by the laboratory manager to all interested parties.

6.2 Report Generation

In general, the chemist performing the analyses will be the person responsible for generating and issuing the analytical reports. In most instances, the laboratory manager will have an opportunity to review reports before issue to clients. Due to the nature of on-site work, however, peer review of reports may not always be feasible. Copies of analytical reports for EPA Methods 8260 and 8270 can be found in Appendix III.

6.3 Data Archives

All pertinent information (raw data, quantitation reports, QA/QC reports and final analytical reports) is archived on backup computer disks and held for a period of ten (10) years.

Analytical Mobile Services, Inc.

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Chain of Custody

Project Name:	**Sampler(s):**	**Date:**
Relinquished by:	**Received by:**	**Time:**

Sample ID	Date	Time	Description	Quantity	8260A	8270B	Other (spec)	Other (spec)	Remarks

Objective:
This protocol describes the procedures for the determination of volatile organics in soil and groundwater samples by Gas Chromatography/Mass Spectrometry. The objective of this protocol is to provide a detailed explanation of work practices adhered to in AMS' mobile analytical laboratory

Applicability:
Laboratory managers and analytical chemists directly involved in the analysis and reporting of environmental samples.

General:
Sample preparation and analysis procedures for all environmental samples will be conducted in a thorough and stepwise manner as indicated by the methods described below for each medium sampled. All personnel performing these analyses must be trained in the procedures and all pertinent AMS standard protocols are to be followed.

These protocols are based upon the following EPA approved methods outlined in "Test Methods for Evaluating Solid Waste," EPA SW-846, 3rd Edition: Method 5030A - "Purge-and-Trap," and Method 8260A - "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique." The individual determinative methods should be referenced for a more detailed explanation of scope, application, interferences, etc. Any changes in EPA or other regulatory procedures will be incorporated during periodic revisions of this SOP.

Equipment:
1) Gas chromatograph/mass spectrometer system - Hewlett-Packard 5890 Series II GC directly coupled to Hewlett-Packard 5972 MS
2) Purge-and-trap device - Tekmar 3000 coupled to Tekmar Precept auto-sampler
3) Column - Supelco VOCOL 60m x 0.25mm ID x 1.5um film thickness
4) Data system - PC with HP MS Chemstation software for acquisition, HP Enviroquant for integration and quantitation, and the NIST 75K Mass Spectral Library database.
5) Drying oven - Fisher Isotemp Standard
6) Syringes - Hamilton Gastight 10-500 uL
7) Assorted glassware including test tubes, volumetric flasks, beakers, vials, and Pasteur pipettes

PROCEDURES:

Standard Preparation:
Stock standard solutions are purchased as certified solutions from Supelco or other reputable vendors. They are stored in bottles with Teflon lined screw-caps in the standards freezer which is kept at -10°C to -20°C and checked frequently for signs of degradation or evaporation, especially prior to use. Standard solutions will have a holding

time of one year. The following standard solutions are used in volatile sample preparation and analysis:

1) Internal standard - Purchased as certified solution from Supelco containing Chlorobenzene-d_5, 1,4-Dichlorobenzene-d_4, 1,4-Difluorobenzene and Pentafluorobenzene at a concentration of 2000 ug/mL. A secondary dilution of internal standard is prepared at a concentration of 50 ug/ml. Each 5 mL sample undergoing analysis is spiked with 5 uL of the internal standard solution, resulting in a concentration of 50 ug/L of each internal standard.

2) GC/MS tuning standard - A 25,000 ug/ml solution of 4-Bromofluorobenzene purchased as a certified solution from Supelco. Diluted to form a secondary standard containing 50 ug/mL. A 50 ug/L BFB standard must be run every 12 hours. The tuning criteria for BFB are as follows:

Mass 50	15 to 40% of mass 95
Mass 75	30 to 60% of mass 95
Mass 95	base peak, 100% relative abundance
Mass 96	5 to 9% of mass 95
Mass 173	less than 2% of mass 174
Mass 174	greater than 50% of mass 95
Mass 175	5 to 9% of mass 174
Mass 176	greater than 95% but less than 101% of mass 174
Mass 177	5 to 9% of mass 176

3) Calibration standards - Calibration standards are prepared at 10, 20, 50, 100, and 200 ug/mL. These concentrations correspond to the working range of the GC/MS system. Each standard contains all analytes for detection by this method plus internal standards and surrogates. The analytes are purchased in certified mixes from Supelco. The %RSD for all levels must not exceed 15% for any compound. If all %RSDs are <15%, the RF is assumed to be constant. If the %RSD >15%, a calibration surve of response ratios versus RF must be plotted.

4) Surrogate standards - Purchased as certified solutions from Supelco. Standard includes 4-Bromofluorobenzene, Toluene-d_8 and Dibromofluoromethane at a concentration of 2000 ug/mL. A secondary solution of 50 ug/mL in methanol is prepared. Each sample for analysis is spiked with 5 uL of the secondary solution. The recovery control limits for aqueous samples are as follows: 4-Bromofluorobenzene 86-115%, Dibromofluoromethane 86-118%, Toluene-d8 88-110%. The recovery control limits for soil samples are as follows: 4-Bromofluorobenzene 74-121%, Dibromofluoromethane 80-120%, Toluene-d8 81-117%..

5) Matrix spike standards - Matrix spike solution is purchased from Supelco. The solution contains Benzene, Toluene, Chlorobenzene, Trichloroethene and 1,1-Dichloroethene at a concentration of 25 ug/mL in methanol.

6) Calibration Check Compounds - Stock standard solution of 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene and Vinyl Chloride at 2000 ug/ml is purchased from Supelco or other respected vendor. A secondary solution of 50 ug/ml is prepared in methanol. A 10 ul aliquot of the secondary solution is added to 5 ml reagent water resulting in a 100 ug/L CCC. The % Drift is then calculated for each CCC. If the % Drift is <20%, the initial calibration is assumed to be valid. CCC injections are required every 12 hours of operation.

7) System Performance Check Compounds - Stock standard solution of Chloromethane, 1,1-Dichloroethane, Bromoform, Chlorobenzene and 1,1,2,2-Tetrachloroethane at 2000 ug/ml is purchased from Supelco or other respected vendor. A secondary solution of 50 ug/ml is prepared in methanol. A 10 ul aliquot of the secondary solution is added to 5 ml reagent water resulting in a 100 ug/L SPCC. The minimum relative response factor must be met for each compound in order for analysis to begin. The minimum relative response factors are as follows: Chloromethane =

0.10, 1,1-Dichloroethane = 0.10, Bromoform = >0.10, Chlorobenzene = 0.30, 1,1,2,2-Tetrachloroethane = 0.30. SPCC must be run every 12 hours along with the CCC.

The following reagents are also used in volatile sample preparation and analysis:

1) Methanol - Fisher Purge and Trap grade
2) Water - organic-free reagent water

All solvents and certified solutions purchased and all standards prepared are recorded in the standard preparation and chemical inventory log book.

Purge-and-Trap:

The purge-and-trap apparatus consists of two primary pieces of equipment; the Tekmar Precept robotic auto-sampler and the Tekmar 3000 purge-and-trap unit. The Precept is capable of holding up to 48 water and/or soil samples and accessing them one at a time in pre-programmed order. The auto-sampler transfers water samples to the 3000 unit directly or can dilute the sample with organic-free reagent water prior to transfer. Soil samples are mixed automatically with organic-free reagent water, heated and purged. The purge gas is transferred directly to the 3000. All internal standards and surrogates are metered in to all samples automatically by the Precept.

GC Operating Conditions:

Carrier gas (Helium) flow rate:	1.0 mL/min
Initial temperature:	35° C, hold for 4 minutes
Temperature program:	35° - 200° C at 4°/min
Final temperature:	200° C, hold until all expected compounds have eluted
Purge:	11 min, 35 ml/min
Desorb:	225° C for 2 min, 20 ml/min
Bake:	225° C for 10 min
Injector temperature:	250 degrees C
Transfer line temperature:	280 degrees C
Source temperature:	approx. 185 degrees C
Scan Range:	45-260 amu
Scan Time:	approx. 2 scans/sec

Initial Calibration:

Calibration must take place using the same sample introduction method that is used to analyze actual samples. Calibration standards are prepared by adding volumes of one or more certified standard mixes to 5 mL of organic-free reagent water. To each calibration standard, 5 uL of internal standard solution is added , resulting in a concentration of 50 ug/L of each internal standard. The calibration standards are then analyzed and the peak area responses are tabulated against the concentration of each compound by the software program. Response factors (RFs) are calculated for each compound. If the percent relative standard deviation (%RSD) of the compound is <15%, the RF is assumed to be constant, and the average RF is used for calculations. If the %RSD is >15%, a calibration curve of response ratios versus RF is plotted.

Daily GC Calibration:

The working calibration is verified on each working day by the measurement of a mid-concentration CCC standard containing all analytes for detection by these methods. If the response for any analyte varies from the predicted response by more then 20%, a new calibration is prepared for that compound.

GC Analysis:

All samples are introduced to the chromatograph via purge&trap. For aqueous samples, a 5 ml portion is removed from the sample container by the Precept robotic auto-sampler and transferred to the fritted sparging tube. Helium is then bubbled through the water sample for eleven (11) minutes. All purge gas is directed to the trap where contaminants are held by the trap packing while the helium carrier gas passes through. Soil samples are purged directly in the Precept robotic auto-sampler. A five (5) gram portion of the soil sample is weighed out and placed into an empty VOA vial. The Precept adds reagent water, internal standards and surrogate standards directly to the vial. This mixture is then purged with helium for eleven (11) minutes and the purge gas is transferred directly to the trap via heated transfer line. High level soil (>1 ppm) is first extracted with reagent grade methanol in a zero head-space container. Sixteen grams of soil are added to a 40 ml VOA vial and then filled to the top with methanol. The vial is then sealed and shaken vigorously for several minutes. A syringe is then used to transfer a small quantity of the methanol into a purge vial filled with 5 ml of reagent water. Helium is then bubbled through the water/methanol solution and carried through to the trap. The purge & trap concentrator then preheats the trap to desorb temperature and waits for a "system ready" signal from the GC. Once the GC is ready, the analytes are desorbed from the trap, transferred to the GC via heated transfer line and the analysis begins.

The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background subtraction, with characteristic ions in the reference mass spectrum. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. Specific guidelines for qualitative identification presented in Method 8260A should be followed. When a compound has been

identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion (quantitation ion). The concentration in the extract is determined by the software program using the average response factor from the initial calibration and the formulas given in Method 8260A.

Quality Control:

The methods require the operation of a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and ongoing analysis of reagent blanks and spiked samples that are subjected to exactly the same analytical procedures as those used on actual samples. AMS' quality control program is outlined in the Quality Assurance Plan (QAP).

The initial demonstration of laboratory capability is encapsulated by the following operations. A quality control reference sample concentrate is prepared containing each analyte at a concentration of 20 ug/mL in methanol. The QC reference sample concentrate is made using stock standards prepared independently from those used for calibration. This is accomplished by the use of Separate Source Standards. QC reference samples are prepared at a concentration of 10 ug/L by adding 2.5 uL of QC reference sample concentrate to each of four 5 mL aliquots of water. The well-mixed samples are then

analyzed according to the methods above. The average recovery and the standard deviation of the recovery is calculated for each analyte using the four results.

Finally, method detection limit (MDL) studies are also required. The procedures for calculating MDLs are taken from 40 CFR 136 App. B. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. A minimum of seven aliquots of water are spiked with all analytes at or near their quantitation limits. The well-mixed samples are then analyzed according to the methods above. The average recovery and the standard deviation is calculated for each analyte using the seven results. The MDL is computed by multiplying the standard deviation by the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom.

Objective:
This protocol describes the procedures for the determination of semi-volatile organics in soil and groundwater samples by Gas Chromatography/Mass Spectrometry. The objective of this protocol is to provide a detailed explanation of work practices adhered to in AMS' mobile analytical laboratory

Applicability:
Laboratory managers and analytical chemists directly involved in the analysis and reporting of environmental samples.

General:
Sample preparation and analysis procedures for all environmental samples will be conducted in a thorough and stepwise manner as indicated by the methods described below for each medium sampled. All personnel performing these analyses must be trained in the procedures and all pertinent AMS standard procedures are to be followed.

These protocols are based upon the following EPA approved methods outlined in "Test Methods for Evaluating Solid Waste," EPA SW-846, 3rd Edition: Method 3510B - "Separatory Funnel Liquid-Liquid Extraction," Method 3550A - "Ultrasonic Extraction," and Method 8270B - "Semi-volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) : Capillary Column Technique." The individual determinative methods should be referenced for a more detailed explanation of scope, application, interferences, etc. Any changes in EPA or other regulatory procedures will be incorporated during periodic revisions of this SOP.

Equipment:
1) Gas chromatograph/mass spectrometer system - Hewlett-Packard 5890 Series II GC directly coupled to Hewlett-Packard 5972 MS with HP 7673B auto-sampler.
2) Column - Hewlett-Packard HP5-MS 30m x 0.25mm ID x 0.25um film thickness.
3) Data system - PC with HP MS Chemstation software for acquisition, HP Enviroquant for integration and quantitation, and the NIST 75K Mass Spectral Library database.
4) Water bath - Fisher Isotemp 10 liter bath
5) pH meter - Fisher Scientific
6) Sonicator - Fisher 550 Sonic Dismembrator
7) Drying oven - Fisher Isotemp Standard
8) Syringes - Hamilton Gastight 10-500 uL
9) Assorted glassware including beakers, filter flasks, graduated cylinders, volumetric flasks, separatory funnels, K-D apparatus, vials, and pipettes.

PROCEDURES:

Standard Preparation:
Standard solutions are purchased as certified solutions from Supelco or other reputable vendors. They are stored in bottles with Teflon lined screw-caps in the standards freezer which is maintained between -10°C to -20°Cand checked frequently for signs of degradation or evaporation, especially prior to use. Standard solutions will have a holding

time of one year. The following standard solutions are used in semi-volatile sample preparation and analysis:

1) Internal standard - Purchased as certified solution from Supelco at a concentration of 2000 ug/mL. Each 1 mL sample extract undergoing analysis is spiked with 20 uL of the internal standard solution, resulting in a concentration of 40 ug/mL of each internal standard.

2) GC/MS tuning standard - Two certified solutions are purchased from Supelco. One contains DFTPP at 2000 ug/mL and the other contains 50 ug/mL each of 4,4'-DDT, pentachlorophenol, and benzidine. 25 uL of the DFTPP solution is added to each 1 mL aliquot of the tuning solution to form a standard containing 50 ug/mL of all four components. This tuning solution must be injected into the chromatograph every 12 hours. The tuning criteria for DFTPP are as follows:

Mass 51	30-60% of mass 198
Mass 68	<2% of mass 69
Mass 70	<2% of mass 69
Mass 127	40-60% of mass 198
Mass 197	<1% of mass 198
Mass 198	Base peak, 100% relative abundance
Mass 199	5-9% of mass 198
Mass 275	10-30% of mass 198
Mass 365	>1% of mass 198
Mass 441	Present but less than mass 443
Mass 442	>40% of mass 198
Mass 443	17-23% of mass 442

In addition, degradation of DDT to DDD and DDE should not exceed 20%. Pentachlorophenol and benzidine should be present at their normal responses and should not exhibit any peak tailing. If these criteria are not met, the injection port should be maintained and the first few inches of the column removed.

3) Calibration standards - Calibration standards are prepared at 20, 40, 80, 120, and 160 ug/mL. These concentrations correspond to the working range of the GC/MS system. Each standard contains all analytes for detection by this method plus internal standards and surrogates. The analytes are purchased in certified mixes from Supelco. The %RSD for all levels must not exceed 15% for any compound. If all %RSDs are <15%, the RF is assumed to be constant. If the %RSD >15%, a calibration surve of response ratios versus RF must be plotted.

4) Surrogate standards - Two surrogate solutions are purchased from Supelco. The acid surrogate solution contains phenol-d6, 2-fluorophenol, and 2,4,6-tribromophenol at a concentration of 10000 ug/mL. The base surrogate solution contains nitrobenzene-d5, 2-fluorobiphenyl, and p-terphenyl-d14 at a concentration of 5000 ug/mL. Limits in aqueous samples are as follows: phenol-d6 (10-94), 2-fluorophenol (21-100), 2,4,6-tribromophenol (10-123), nitrobenzene-d5 (35-114), 2-fluorobiphenyl (43-116) and p-terphenyl-d14 (33-141). Limits in soil samples are as follows: phenol-d6 (24-113), 2-fluorophenol (25-121), 2,4,6-tribromophenol (19-122), nitrobenzene-d5 (23-120), 2-fluorobiphenyl (30-115) and p-terphenyl-d14 (18-137).

5) Matrix spike standards - Two matrix spike solutions are purchased from Supelco. The acid matrix spike solution contains pentachlorophenol, phenol, 2-chlorophenol, 4-chloro-3-methylphenol, and 4-nitrophenol at a concentration of 2000 ug/mL. The base matrix spike solution contains 1,2,4-trichlorobenzene, acenaphthene, 2,4-dinitrotoluene, pyrene, n-nitroso-di- n-propylamine, and 1,4-dichlorobenzene at a concentration of 1000 ug/mL. Recovery criteria for matrix spike compounds

4-chloro-3-methylphenol (40.8-127.9), and 4-nitrophenol (13.0-106.5), 1,2,4-trichlorobenzene (57.3-129.2), acenaphthene (60.1-132.3), 2,4-dinitrotoluene (47.5-126.9), pyrene (69.6-100.0), n-nitroso-di-n-propylamine (13.6-197.9) and 1,4-dichlorobenzene (37.3-105.7).

The following reagents are also used in semi-volatile sample preparation and analysis:

1) Methylene chloride - Fisher Optima
2) Water - organic-free reagent water
3) Sodium sulfate - Fisher anhydrous, granular, certified ACS
4) Sodium hydroxide - Fisher solution 50% w/w
5) Sulfuric acid - Fisher reagent, certified ACS

All solvents and certified solutions purchased and all standards prepared are recorded in the standard preparation and chemical inventory log book.

Extraction of Aqueous Method Blank

Using a 1 liter graduated cylinder, 1 liter of organic-free reagent water is measured and then transferred to a separatory funnel. With a 25 ul syringe, 10 ul of acid surrogate standard and 20 ul of base surrogate standard are added to all samples, spikes, and blanks. For the sample in each analytical batch selected for spiking, 50 ul of acid matrix spike and 100 ul of base matrix spike are added with a 100 ul syringe. These amounts result in a final concentration of 100 ug/mL of each surrogate and matrix spike compound.

The pH of the sample is checked with a pH meter and then adjusted to <2 with sulfuric acid. Approximately 60 mL of methylene chloride is then added to the separatory funnel. The funnel is sealed and shaken vigorously for 1-2 minutes with periodic venting to release excess pressure. The funnel is then placed on a ring stand and the layers are allowed to separate for 10 minutes, after which the solvent extract is collected in a 250 mL beaker. The extraction is repeated twice using fresh portions of solvent. The pH of the sample is adjusted to >11 with sodium hydroxide solution and serially extracted three times as above. These extracts are collected in a separate beaker.

Extraction of Liquids:

Using a 1 liter graduated cylinder, 1 liter of sample is measured and then transferred to a separatory funnel. With a 25 ul syringe, 10 ul of acid surrogate standard and 20 ul of base surrogate standard are added to all samples, spikes, and blanks. For the sample in each analytical batch selected for spiking, 50 ul of acid matrix spike and 100 ul of base matrix spike are added with a 100 ul syringe. These amounts result in a final concentration of 100 ug/mL of each surrogate and matrix spike compound.

The pH of the sample is checked with a pH meter and then adjusted to <2 with sulfuric acid. Approximately 60 mL of methylene chloride is then added to the separatory funnel. The funnel is sealed and shaken vigorously for 1-2 minutes with periodic venting to release excess pressure. The funnel is then placed on a ring stand and the layers are allowed to separate for 10 minutes, after which the solvent extract is collected in a 250 mL beaker. The extraction is repeated twice using fresh portions of solvent. The pH of the sample is adjusted to >11 with sodium hydroxide solution and serially extracted three times as above. These extracts are collected in a separate beaker.

Concentration of Liquid Extracts:
A Kuderna-Danish (K-D) concentrator is assembled by attaching a 10 mL concentrator tube to a 250 mL evaporation flask. The extracts are dried by adding sodium sulfate until all water is removed. The dried extracts along with beaker washings are combined and vacuum filtered through a 0.45 um membrane that is attached to a 500 mL filtration flask. The extract is then transferred to the K-D concentrator, along with flask washings. A boiling chip is added to the flask and a three ball Snyder column is attached. The column is pre-wetted by adding methylene chloride to the top of the column. The entire K-D apparatus is then placed on a water bath set at approximately 70 degrees C. When the liquid level reaches the lower part of the concentrator tube, the K-D apparatus is removed from the water bath and allowed to drain. After the apparatus has cooled, the Snyder column is removed; the flask is rinsed with methylene chloride and then removed. A clean boiling chip is added and a two ball micro-Snyder column is attached and pre-wetted. The apparatus is placed back in the water bath until the liquid volume reaches 1 mL. The final extract is then pipetted into a vial with a Teflon lined screw-cap and labeled appropriately.

Extraction of Soil Blank
A known clean soil sample is mixed thoroughly with a spatula. The percent dry weight is determined so that results may be reported on a dry weight basis. In order to determine the dry weight, approximately 10 g of the sample is weighed into a tared crucible and dried overnight at 105 degrees C. The percent dry weight is obtained by dividing the weight of the remaining dry sample by the amount originally used.

30 g of sample is weighed into a 250 mL beaker. Sodium sulfate is added until the mixture becomes a free flowing powder. With a 25 ul syringe, 10 ul of acid surrogate standard and 20 ul of base surrogate standard are added to all samples, spikes, and blanks. For the sample in each analytical batch selected for spiking, 50 ul of acid matrix spike and 100 ul of base matrix spike are added with a 100 ul syringe. These amounts result in a final concentration of 100 ug/mL of each surrogate and matrix spike compound. Approximately 100 mL of methylene chloride is added immediately. The bottom surface of the sonicator disrupter horn is placed between the surface of the solvent and the sediment layer. The sample is extracted ultrasonically for 3 minutes at full power with pulsing every second. The extract is decanted and filtered through a 0.45 um membrane that is attached to a 500 mL filtration flask. The extraction is repeated twice with fresh solvent. After the final extraction, the entire sample is poured into the filter reservoir along with beaker washings and vacuum filtered until all visible solvent is removed from the sample.

Extraction of Soils and Sediments:
After decanting any water layer, a soil or sediment sample is mixed thoroughly with a spatula and foreign objects are discarded. The percent dry weight is determined so that results may be reported on a dry weight basis. In order to determine the dry weight, approximately 10 g of the sample is weighed into a tared crucible and dried overnight at 105 degrees C. The percent dry weight is obtained by dividing the weight of the remaining dry sample by the amount originally used.

30 g of sample is weighed into a 250 mL beaker. Sodium sulfate is added until the mixture becomes a free flowing powder. With a 25 ul syringe, 10 ul of acid surrogate standard and 20 ul of base surrogate standard are added to all samples, spikes, and blanks. For the sample in each analytical batch selected for spiking, 50 ul of acid matrix spike and 100 ul of base matrix spike are added with a 100 ul syringe. These amounts result in a final concentration of 100 ug/mL of each surrogate and matrix spike compound. Approximately 100 mL of methylene chloride is added immediately. The bottom surface of the sonicator disrupter horn is placed between the surface of the solvent and the sediment layer. The sample is

extracted ultrasonically for 3 minutes at full power with pulsing every second. The extract is decanted and filtered through a 0.45 um membrane that is attached to a 500 mL filtration flask. The extraction is repeated twice with fresh solvent. After the final extraction, the entire sample is poured into the filter reservoir along with beaker washings and vacuum filtered until all visible solvent is removed from the sample.

Concentration of Soil Extracts:

A Kuderna-Danish (K-D) concentrator is assembled by attaching a 10 mL concentrator tube to a 250 mL evaporation flask. The filtered extract is transferred to the concentrator, along with flask washings. A boiling chip is added to the flask and a three ball Snyder column is attached. The column is pre-wetted by adding methylene chloride to the top of the column. The entire K-D apparatus is then placed on a water bath set at approximately 70 degrees C. When the liquid level reaches the lower part of the concentrator tube, the K-D apparatus is removed from the water bath and allowed to drain. After the apparatus has cooled, the Snyder column is removed; the flask is rinsed with methylene chloride and then removed. A clean boiling chip is added and a two ball micro-Snyder column is attached and pre-wetted. The apparatus is placed back in the water bath until the liquid volume reaches 1 mL. The final extract is then pipetted into a vial with a Teflon lined screw-cap and labeled appropriately. Further clean-up of the extracts will not be required in most situations. If it is deemed necessary, an appropriate sample clean-up procedure (as outlined in SW-846) will be performed prior to introducing the extract into the GC/MS.

GC/MS Operating Conditions:

Mass range:	35-500 amu
Scan time:	1.6 scan / sec
Initial temperature:	50 degrees C, hold for 4 minutes
Temperature program:	50-300 degrees C at 10 degrees / min
Final temperature:	300 degrees C, hold until benzo(g,h,i)perylene has eluted
Injector temperature:	280 degrees C
Transfer line temperature:	300 degrees C
Source temperature:	approx. 175 degrees C
Injector:	splitless
Sample volume:	1 uL
Carrier gas:	Helium at 1 mL / min

These conditions apply to all runs except tuning runs, which use a shorter run time starting at 100 degrees C, holding for 1 minute and ramping at 15 degrees / minute to 300 degrees C.

Initial Calibration:

The system is hardware-tuned until the criteria in Table 3 of Method 8270B are met for a 50 ng injection of the DFTPP tuning standard. Background subtraction is used only to eliminate column bleed or instrument background ions. The tuning standard is also used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not be excessive. The sum of the peak areas for the breakdown products divided by the sum of the three peak areas should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, which are comparable to that of DFTPP. If degradation of any compound is excessive, the injection port is maintained and several inches removed from the front of the column.

The internal standards (ISTD) selected should permit most of the components of interest to have retention times of 0.80-1.20 relative to one of the ISTD. 1 uL of each of the five calibration standards is analyzed.

Response factors (RF) for each compound relative to the appropriate ISTD are calculated and tabulated against concentration by the software program. A system performance check is performed to ensure that minimum average RF are met before the calibration is used. The System Performance Check Compounds (SPCC) are n-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitro-phenol, and 4-nitrophenol. The minimum average RF for these compounds is 0.050. The percent relative standard deviation (%RSD) of the response factors for the 13 Calibration Check Compounds (CCC) listed in Table 4 of Method 8270B must be less than 30%. If the %RSD of any CCC is 30% or greater, then the chromatographic system is too reactive for analysis to begin; the injection port is maintained and several inches removed from the front of the column. If all of the SPCC and CCC meet these criteria, then the initial calibration is deemed to have passed, and analysis of samples may begin.

Daily GC/MS Calibration:

Prior to the analysis of samples, the GC/MS tuning standard must be analyzed. A 50 ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 3 of Method 8270B. These criteria must be demonstrated during each 12 hour shift. A calibration standard at mid-concentration containing all semi-volatile analytes, including required surrogates, must also be analyzed every 12 hours during analysis. For each SPCC in the daily calibration, a minimum response factor of 0.050 must be obtained. After the system performance check is met, CCC are used to check the validity of the initial calibration. If the percent drift of the response factor for each CCC is less than or equal to 20%, the initial calibration is still valid and sample analysis may begin. If any one CCC or SPCC does not meet criteria, then corrective action must be taken. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration must be generated. If the CCC or SPCC which fail in the daily calibration are not required analytes, then all required analytes must meet the 20% drift criterion before sample analysis can begin.

GC/MS Analysis:

The 1 mL extract obtained from sample preparation is spiked with 20 uL of the internal standard solution just prior to analysis. The sample is then analyzed by GC/MS using a 30 m x 0.25 mm silicone-coated fused-silica capillary column. The 1 uL injected contains 100 uL of base and acid surrogates. The GC/MS operating conditions used are specified above. If the concentration of any analyte exceeds the initial calibration range of the GC/MS system, extract dilution is performed. Additional ISTD is added to the diluted extract to maintain the required 40 ug/mL of each internal standard. The diluted extract is then reanalyzed. Extracts are stored in the sample refrigerator, protected from light, in screw-cap vials with Teflon lined septa.

The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background subtraction, with characteristic ions in the reference mass spectrum. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. Specific guidelines for qualitative identification presented in Method 8270B should be followed. When a compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion (quantitation ion). The concentration in the extract is determined by the software program using the average response factor from the initial calibration and the formulas given in Method 8270B.

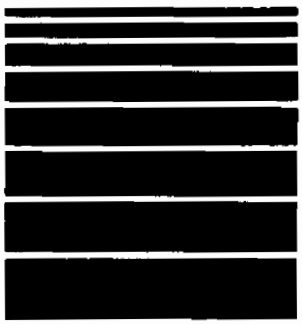
Quality Control:

The methods require the operation of a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and ongoing analysis of reagent blanks and spiked samples that are subjected to exactly the same analytical procedures as those used on actual samples. A method blank, matrix spike and matrix spike duplicate must be analyzed for each analytical batch. If the laboratory only analyzes one to ten samples per month, one spiked sample is required. AMS' quality control program is outlined in the Quality Assurance Plan (QAP).

The initial demonstration of laboratory capability is encapsulated by the following operations. A quality control reference sample concentrate is prepared containing each analyte at a concentration of 100 ug/mL in methanol. The QC reference sample concentrate is made using stock standards prepared independently from those used for calibration. This is accomplished by the use of Separate Source Standards. QC reference samples are prepared at a concentration of 100 ug/L by adding 1 mL of QC reference sample concentrate to each of four 1 L aliquots of water. The well-mixed samples are then analyzed according to the methods above beginning with extraction of the samples. The average recovery and the standard deviation of the recovery is calculated for each analyte using the four results. The results are then compared to the acceptance criteria found in Table 6 of Method 8270B. When one or more of the analytes tested fail at least one of the acceptance criteria, the test is repeated for those analytes.

Finally, method detection limit (MDL) studies are also required. The procedures for calculating MDL are taken from 40 CFR 136 App. B. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

A minimum of seven aliquots of water are spiked with all analytes at or near their quantitation limits listed in Table 2 of Method 8270B. The well-mixed samples are then analyzed according to the methods above beginning with extraction of the samples. The average recovery and the standard deviation is calculated for each analyte using the seven results. The MDL is computed by multiplying the standard deviation by the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom.



COMPUCHEM
ENVIRONMENTAL CORPORATION

QUALITY ASSURANCE PLAN

1160-I

1.0 Title and Signatures

CompuChem Environmental Corporation Generic Quality Assurance Plan

CompuChem Environmental Corporation
3306 Chapel Hill Nelson Highway
PO Box 14998
RTP, NC 27709-4998

Responsible Officials:

Gerard C. Verkerk, President and Chief Executive Officer

Signature: _____ Date: 4/4/94

Robert E. Meierer, Vice President, General Manager

Signature: _____ Date: 4/4/94

Quality Assurance Officer:

Linda Fowler, Manager of Quality Assurance

Signature: Linda Fowler Date: 4/4/94

Plan Coverage:

Statement of Policy
QA Management, Personnel, and Training
Analytical Procedures
Sample Receipt and Custody
Sample and Hardcopy Data Custody and Control
Calibration/Frequency
Document Control Procedures
Data Reduction, Evaluation, and Reporting
QC Samples and Documentation
Performance and System Audits/QA Audits
Equipment and Instrument Maintenance
QA Objectives for Measurement
Corrective Action and Documentation
QA Reports to Management
Facilities and Safety
Method Development
Procurement Control

2.0 Table of Contents

3.0 Statement of Policy

The management of CompuChem Environmental Corporation (hereafter referred to as CompuChem) is fully and firmly committed to the quality assurance (QA) program described in this QA Plan. Each director, manager, and supervisor, as well as their staff members, as assigned in accordance with this plan, is obligated to comply with its stated requirements, responsibilities, and objectives.

The primary QA objective is to develop and implement procedures for sample receiving, chain-of-custody, sample preparation, laboratory analysis, data verification and evaluation, and reporting that will provide data that are legally defensible. Key aspects of these procedures are described in this QA Plan, while specific details are included in the laboratory's and in the QA department's standard operating procedures (SOPs).

The QA program is maintained and expanded or modified as necessary to ensure that all reported data are of uncompromised quality. To determine whether QA objectives are met, sufficient quality control (QC) is generated to evaluate precision, accuracy, and completeness, and, when possible, a statement regarding representativeness and comparability is provided.

This QA Plan complies with the requirements, guidelines, and specifications found in the following documents:

U.S. EPA. (1980). *Guidelines and specifications for preparing quality assurance program plans.* QA Office of Research and Development, QAMS-004/80.

U.S. EPA. (1980). *Interim guidelines and specifications for preparing quality assurance project plans.* Office of Monitoring Systems and Quality Assurance/ Research and Development, QAMS-005/80.

U.S. Department of Energy. (1990). *Requirements for quality control of analytical data.* HWP-65/R1.

American National Standards Institute/American Society of Mechanical Engineers. (1989). *Quality assurance program requirements for nuclear facilities.* NQA-1.

NEESA. (1988). *Sampling and chemical analysis quality assurance requirements for the Navy installation restoration program.* NEESA 20.2-047B.

American National Standards Institute/ASQC. (1991). *Quality assurance program requirements for environmental programs.* ANSI/ASQC-E4-19xx (formerly EQA-1).

ES/ER/TM-16. (1992). *Requirements for quality control of the analytical data for the environmental restoration program.* ES/ER/TM-16.

There are several supplements to this QA Plan which are described in the various sections to which they are applicable. To order these supplements simply complete and detach the form below and return it to the CompuChem address listed in Section 1.0, Title and Signatures.

CompuChem Environmental Corporation QA Plan Supplements Order Form

Please check those supplements to CompuChem Environmental Corporation's Generic QA Plan that you would like to receive, then fill in the destination name and address as completely and legibly as possible. Mail directly to CompuChem Technical Communications Department. Allow 2-4 weeks for delivery.

☐	Supplement A	Corporate Organization and Resumes of Key Management Personnel
☐	Supplement B	Laboratory Experience Record for Technical Staff
☐	Supplement C	Laboratory Equipment Inventory
☐	Supplement D	Calculations Used in Data Reduction
☐	Supplement E	Method Detection Limit Studies

DESTINATION:

Name: _____

Company/Organization: _____

Department: _____ Mailstop: _____

Street Address: _____

_____

_____

City: _____State: _____

PO Box : _____

4.0 Quality Assurance (QA) Management/Personnel Qualifications and Training

With over 170 employees, CompuChem offers the scientific and technical expertise needed to fulfill the analytical and informational needs of our customers. In addition to our experienced analytical laboratory personnel (with specialized skills in organics, inorganics, and radiological analyses), CompuChem has a computer systems staff that plans, develops, and implements software systems for data management and sample scheduling and control. To ensure that CompuChem meets the analytical needs of clients, customer service representatives (account administration) are assigned to each account, and act as a liaison between the customer and the laboratory. A program of project management teams has been implemented to more effectively serve our customers, particularly when a project requires an additional level of oversight. Senior individuals have been identified as technical project managers who lead teams consisting of customer service individuals, sales personnel, and support staff as well as a corporate sponsor. The executive committee participates in weekly meetings with the project management teams to increase communication and awareness, and to continually improve our responsiveness to client needs. When a project management team is assigned, the project manager or the account administrator is the internal customer representative and acts as the primary contact for the customer. Unique specific customer project requirements are communicated to the laboratory through a written project profile sheet (PPS) prepared by the project management team. CompuChem has established a rapport withthe U.S. Environmental Protection Agency (EPA) Program Office in Washington, DC, through our administrative project officer and our technical project officer located in our region (IV). The CEO and senior management are firmly committed to continuous improvement and customer satisfaction and have implemented a process of Total Quality Management (TQM) within the organization.

The following section describes the operational and functional responsibilities of key laboratory personnel, including activities that relate to product and process quality. Also, the roles and responsibilities of the Quality Assurance department and its organizational relationship to laboratory management are identified. Refer to Figures 4-1 through 4-2 for an overview of these relationships.

Organizational charts listing names of those holding the positions and detailed resumes of key technical personnel are available on request as Supplement A to this QA Plan. See Section 3.0 for ordering information. QA department staff, who operate independently of production areas, monitor and audit all laboratory units. All QC criteria are documented, and compliance is verified at each level of laboratory data review.

Standard Operating Procedures (SOPs) for in-lab data evaluation and independent QA auditing describe the details of these quality control functions and associated oversight activities. The QA department is responsible for, among other things, verifying the integrity of these functions and documenting performance for lab management to review.

4.1 Assignment of Responsibilities

The main objectives of CompuChem's QA program are to assure that

- our laboratories generate data of known quality
- data quality meets or exceeds all QA/QC criteria
- the records necessary to document laboratory performance are maintained

The QA department monitors sample processing from initial order entry through the analytical system and to the final data report through a series of audit activities. QA monitors compliance with laboratory SOPs and established good laboratory practices. The QA department is responsible for providing feedback to management and identifying and implementing policies to improve quality. The success of the program depends on the capabilities of those who carry it out. Following are brief summaries of the responsibilities and the authority of each of the QA staff positions.

Vice President, General Manager CompuChem Environmental Corporation

To be certain that the laboratory achieves all QA program objectives, the Vice President General Manager (VPGM) monitors and directs the quality activities of QA department and laboratory personnel. The VPGM, with input from the QA department staff, establishes laboratory policies as needed for activities affecting quality. The VPGM adheres to the procedures and requirements set forth in the QA Plan. The VPGM reports directly to the corporation's Chief Executive Officer (CEO). The VPGM and senior QA staff are responsible for overseeing QA of all laboratory operations. The VPGM and the QA department manager have the authority to terminate nonconforming work at any time.

Additional responsibilities and duties related to the quality program include:

- monitoring the QA program as documented in the QA Plan and ensuring that all elements are carried out as written
- evaluating the effectiveness of quality management systems and reporting evaluations to management and the CEO
- developing and implementing new QA programs, including statistical procedures, additional QC measures, and new methods validation, etc.
- maintaining current documentation of all measurement procedures routinely used in the laboratories, including those used by subcontractors
- implementing or modifying analytical methods to conform with recognized standards and/or GLPs, including alteration of analysis/procedure codes used by the Laboratory Information Management System (LIMS)

- having final authority to terminate or alter any incorrect or improper analytical or measurement procedure to conform to requirements of the QA Program Plan (QAPP)
- training, directing, and qualifying personnel in specified laboratory QC and analytical procedures or designating qualified individuals to do so
- reviewing and advising lab management on requirements and applicability of specific Quality Assurance Project Plans (QAPjPs), new statements-of-work (SOWs), RFQs, RFPs, IFBs, and other contract-related issues
- directing the activities of the QA department and the Technical Communications department, as well as the Systems and Laboratory Automation department staff and the research and development staff

Manager of Quality Assurance

The manager of QA reports to the VPGM and is organizationally and functionally independent of all personnel directly involved in the laboratory operations. The manager of QA is primarily responsible for overseeing and directing the activities of the QA staff, consisting of QA specialists I, II, and III, and clerical staff. The VPGM, the manager of QA, and all QA specialists have authority to approve data. The final technical reviewer has the authority for final data approval. The manager of QA and the VPGM have the authority to terminate nonconforming work at any time.

Additional responsibilities and duties include:

- providing QA reports to management
- overseeing the laboratory's participation in external QA/QC programs
- coordinating external (on-site) and internal QA/QC audits or inspections
- reviewing and approving laboratory-generated data qualifying notices
- writing QA Notices that are used to document exceptions to QC acceptance criteria or other matters affecting data usability or interpretation for inclusion in data packages
- providing training to QA and laboratory staff
- periodically informing management of the status of the QA Plan
- providing assistance on special projects as required by the VPGM

- overseeing all subcontractor QA programs, including administering and reviewing their proficiency studies and conducting on-site audits
- developing and implementing new QA programs, including statistical procedures, additional QC measures, and new methods validation, etc.
- conducting scheduled or unannounced audits and inspections, reporting findings to management and, when needed, ensuring that corrective action is taken
- seeking out and evaluating new ideas and current developments in the field of QA and recommending ways to apply them where advisable
- having final authority to terminate or alter any incorrect or improper analytical or measurement procedure to conform to requirements of the QAPP
- training, directing, and qualifying personnel in specified laboratory QC and analytical procedures or designating qualified individuals to do so
- reviewing and advising lab management on requirements and applicability of specific Quality Assurance Project Plans (QAPjPs), new statements-of-work (SOWs), RFQs, RFPs, IFBs, and other contract-related issues
- reviewing customer problem resolution reports for out-of-control events and verifying that remedial action has been taken to restore control
- assuring that subcontractor laboratories are complying with the QA program
- serving as point-of-contact for exchange of QA/QC information and approving release of QA/QC information
- assuming certain responsibilities of the VPGM, if necessary

Quality Assurance Department Staff

The QA department staff are responsible for carrying out quality activities as directed by the manager of QA and the VPGM. The QA staff are organizationally and functionally independent of all personnel directly involved in the laboratory operations.

Additional responsibilities and duties of the QA department staff include:

- ensuring that the laboratories meet all quality requirements as documented in the QA Plan, as well as those documented in the specific QA and laboratory SOP manuals

- auditing and spot-checking work in process for quality and completeness
- providing deviation/exception reports to laboratory managers and the VPGM regarding out-of-control analyses and providing recommendations for corrective action
- overseeing corrective action as required
- generating, analyzing, and documenting QA/QC data (Much of the QC data is generated by the laboratory staff in the normal course of producing analytical data, or by using the LIMS during data acquisition or data entry.)
- based upon laboratory performance statistics and/or SOW requirements establishing and updating control limits using QC data from routine sample analyses
- providing information and documentation for internal/external audits or inspections
- functioning as a liaison between the QA manager/VPGM and personnel within the laboratories
- communicating QA program objectives and requirements to clients and external auditors
- reinforcing good laboratory practices within the laboratory
- communicating any quality concerns to the QA manager/VPGM
- communicating any safety concerns to the chemical hygiene or radiation safety officer
- reviewing and approving performance evaluation (PE) and proficiency testing (PT) sample data
- reviewing PE and PT scores, coordinating lab personnel review of unacceptable PE/PT scores and associated data, and assembling findings into a unified document for response to certifying agencies
- initiating and documenting corrective action (if necessary) related to audit deficiency reports or unacceptable PE/PT scores
- introducing internal single and double "blind" PE samples into the LIMS and reporting performance to management

- auditing the documentation of approval and traceability of all standards to National Institute of Standards and Technology (NIST), U.S. EPA or other certified source

- auditing the documentation of calibration traceability of all thermometers, balances and Class-S weights used in daily calibrations

- exercising control of purchased items known to affect quality through evaluation and approval on a per lot basis

- conducting routine self-inspections, including performance audits, system audits, and summarizing findings in reports to management

Technical Communications Staff

The Technical Communications staff consists of trained technical writers who design, implement, and maintain various technical communication systems within CompuChem. The supervisor of Technical Communications reports directly to the VPGM. Specific responsibilities include:

- writing, editing, and revising SOPs, as well as enforcing proper document control, maintaining historical records of SOP revisions, and distributing SOPs to laboratory stations

- designing, producing, and issuing logbooks and runlogs through CompuChem's Laboratory Logbook Control System (LLCS)

- revising and distributing CompuChem's QAPP, selected program-specific QAPPs, and selected QA Project Plans (QAPjPs)

- initiating and maintaining laboratory certifications by state and federal agencies

- writing and designing training program media for laboratories

- managing all employee training files, ensuring that training documentation is received and filed, and that completion of training events is documented in CompuChem's electronic training information database

- teaching in-house technical and business communication courses to ensure quality of written communication

- managing an in-house technical library and acting as information specialists for technical staff

Laboratory Personnel and Management

A variety of QC functions and duties directly or indirectly affecting data quality are performed by laboratory personnel and management.

Key responsibilities of this nature include:

- assuring compliance with methods and SOPs as directed by the VPGM
- verifying that all instruments meet calibration and tuning requirements
- identifying, initiating, documenting, and completing corrective action requirements
- performing scheduled, routine preventive maintenance of instruments or overseeing work done under service contracts
- following good laboratory practices and recommendations of the QA department for improving quality and safety
- performing and documenting action steps based on established QC acceptance criteria
- providing adequate and documented training of personnel
- performing various levels of data review to evaluate QC acceptance criteria and verify client and contract compliance

4.2 QA Communications

The QA department communicates with other areas of the laboratory and to management via several different types of reports. The VPGM and the QA staff also distribute interoffice memoranda to appropriate laboratory management detailing the results of internal and external audits, blind interlaboratory proficiency studies, blind internal proficiency studies, and deficiency reports/ corrective action needs. Good laboratory practices and successful performance on various studies and audits are also reinforced through these memoranda.

4.3 QA Program Assessment

The VPGM and the QA department staff conduct periodic assessments of the QA program. Based on these assessments, a written status report of QA activities and progress is forwarded to management. The following items are addressed in these reports; most are addressed in the quarterly and the monthly QA activities reports to management as described in Section 16.0. A quarterly report of the effectiveness of the quality management programs is prepared by the VPGM and reported to the President and CEO.

- status of or changes to the QAPP
- status of QAPjPs, if any

- measures of data quality
- significant QA obstacles, accomplishments, and recommendations
- results of performance audits
- results of system audits
- status of QA requirements for contracts
- summary of QA training (internal and external QA/QC seminars and courses)
- overall effectiveness of the QA program

4.4 Personnel Qualifications

CompuChem, located in Research Triangle Park, NC, and within minutes of three major university campuses, is ideally positioned for recruiting both scientists and experienced professionals with degrees in their fields. Many applied science graduates join the organization as entry-level technicians, and progress through extensive training into senior chemist, data review/verification, and QA positions. In most technician positions and instrument operator positions, the training period lasts from six months to one year, depending on the level of experience required and complexities of the position/instrumentation.

The U.S. EPA has set forth requirements for qualifications of technical personnel involved in analyzing EPA samples. EPA requires the following experience levels for technical personnel who analyze EPA samples. As Supplement B to this QA Plan, the Laboratory Experience Record (LER) demonstrates, CompuChem's laboratory staff exceed these requirements.

ORGANICS:

- Gas chromatograph/ mass spectrometer (GC/MS) operators independently performing work on EPA contracts must each have at least one year of experience in analyzing EPA samples.

- Mass spectral interpretation specialists performing work on EPA contracts must have at least two years of experience ("experience" means more than 50 percent of the personnel's productive work time) in the interpretation of mass spectra gathered in GC/MS analysis.

- Extraction and concentration specialists performing work on EPA contracts must have at least one year of experience in preparing extracts from environmental or hazardous waste samples.

- Pesticide residue analysis experts performing work on EPA contracts must have at least two years of gas chromatography (GC) experience in organochlorine pesticide residue and PCB analysis, and in interpreting GC chromatograms.

INORGANICS:

- Inductively coupled plasma (ICP) spectroscopists responsible for work under EPA contracts must have at least two years of experience in the operations of the ICP on environmental samples. ICP operators must have at least one year of experience.

- Flameless atomic absorption (AA) operators responsible for the work on EPA contracts must have at least one year of experience operating and maintaining AA instrumentation for graphite furnace and cold vapor analysis of environmental samples.

- Inorganic sample preparation specialists performing sample preparation for EPA contracts must have at least one year of experience in sample preparation in an analytical laboratory.

- Classical inorganic techniques analysts (cyanide analyst) responsible for work on EPA contracts must have at least one year of experience with classical chemistry laboratory procedures.

CompuChem's technical personnel meet or exceed these requirements in all cases. Resumes of technical personnel are available on request as Supplement A to this QA Plan. See Section 3.0 for ordering information.

Figure 4-1 **Organization of CompuChem Environmental Corporation**

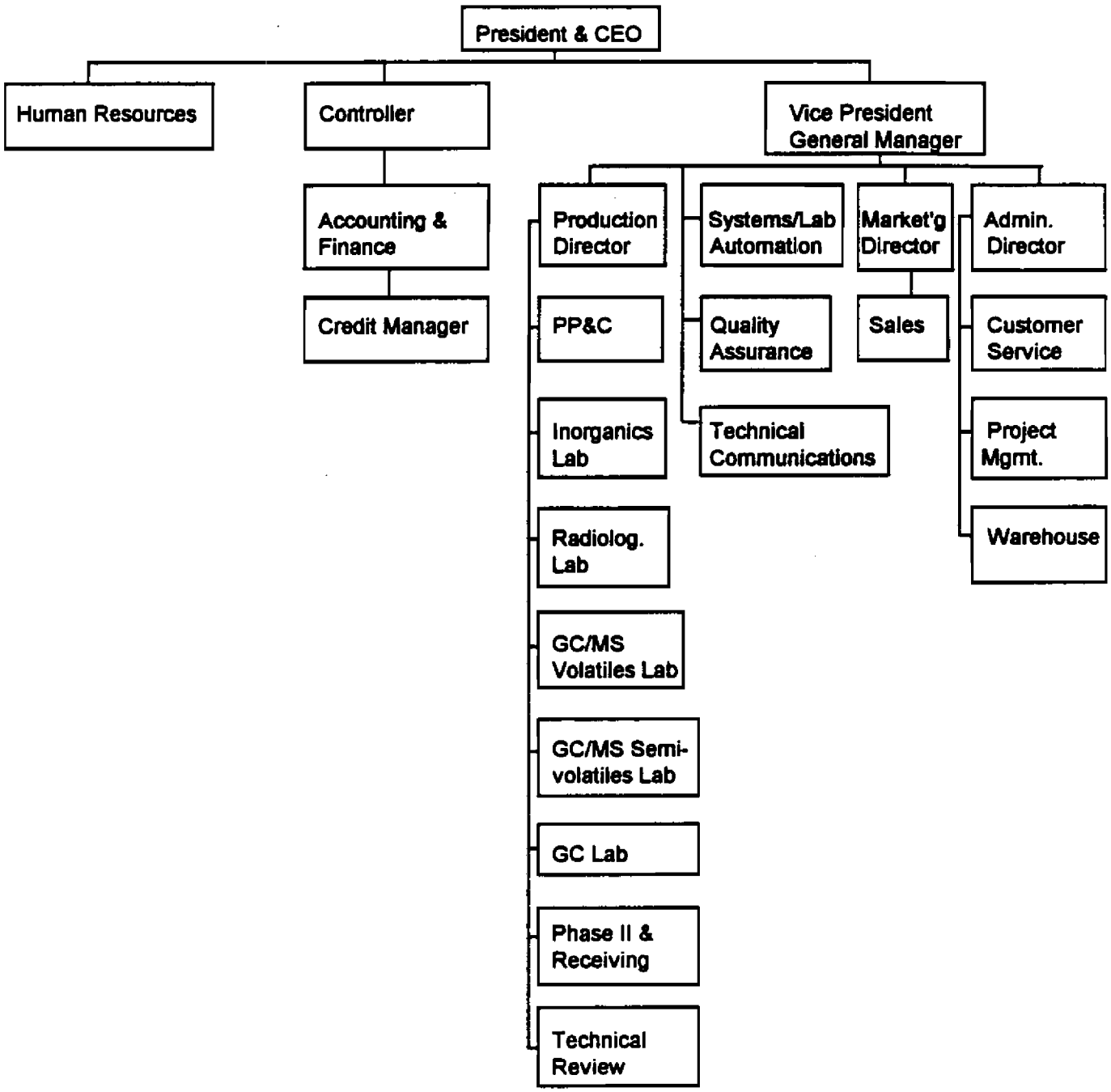


Figure 4-2. QA Oversight in Laboratory Operations

Responsible Section	Lab Operations Processes	Independent QA Oversight
Marketing/order entry	Client/lab interface Analytical requirements determined Order entered into LIMS Forecast of internal workload made	•Interface between lab and external auditors •Audit percentage of data after release •Perform SOP compliance auditing •Generate QA reports to management
Sample Receiving/Control	Sample coolers unloaded and inspected Receiving information entered into LIMS Unique LIMS sample ID assigned Chain-of-Custody (COC) and Sample Tracking worksheets processed Samples stored in refrigerator	P
Standards Lab	Analytical standards prepared Purity and traceability verified	
Sample Preparation Lab	Sample prepared for analysis Glassware prepared Sample preparation worksheets completed	P
Instrumentation Lab(s)	Sample analyzed Data worked up Primary and secondary data review done Routine instrument maintenance performed	•Establish QA/QC criteria •Review contract, work plan,QAPP •Oversee proficiency testing programs
Report Preparation	Data reduction done Word processing done Proofreading done Document deliverables checked	P
Final Technical Review	Data verified and approved Data qualified/flagged if needed Associated QC sample data reviewed Case narrative/summary report written Technical inquiries answered	P
Report Data		

4.5 Training

CompuChem's training program is administered by the Human Resources (HR) department and applies to all full-time employees, and also to any temporary and part-time employees that support a full-time function. All job functions are fully described in formal job descriptions, which are kept on file in HR. To be hired or promoted, an employee must meet all job description requirements. Training checklists are used to document and certify that all position requirements are met. At that time, an employee can be hired or promoted, depending on company needs. All hiring and subsequent changes in personnel status are documented through the use of the Personnel Action Form (PAF).

Various training programs are provided for employees new to a position, and the training records are maintained in the individual's permanent training files, which are currently maintained by the Technical Communications department. Performance is measured through indicators such as precision and accuracy in spike samples, surrogate recoveries and contamination, as well as in productivity or in error rate tabulations. If an employee fails to maintain acceptable performance standards, retraining and recertification must be documented before the employee may work independently. Hardcopy and electronic training records are maintained for each employee, and include dates, locations, credit hour value, grades earned, and proof of completion for each training event completed by each employee.

The job description requirements for each position, and within a "job family progression" are incorporated into the training checklists for each department. The checklists are used to document and certify that all requirements are met before an individual is hired or promoted.

The training checklists (and job descriptions) include minimum acceptable levels of formal education, training, and prior experience. Also included, if applicable, are special requirements for certifications, job-related aptitude tests (e.g., typing or data entry) or licenses. Some contracts or client agreements specify minimum qualifications for certain technical, administrative, computer, and management positions. Additionally, certain positions require auxiliary training, including viewing of training videotapes, on-site training classes, or off-site attendance of specialized training or certification courses. These requirements are identified in the training checklists and job description in such instances. Independently, the Technical Communications department maintains a Laboratory Experience Record (Supplement B to this QA Plan), which chronicles the number of months of experience of laboratory personnel.

All positions directly or indirectly affecting quality of data, data reports, or other customer products or services must be directly supervised during the initial orientation and training period. This period varies in length of time depending upon the nature of the position and specific qualifications of the person in the position. For certain jobs requiring specific experience, training, or certification, the incumbent is known as a *trainee* until these minimum qualifications are met. Any work performed by a trainee is directly supervised, and any worksheets, forms, or other analytical data, whether "deliverable" or not, must be reviewed and counter-signed by the supervisor or designated senior staff member.

The formal training program at CompuChem also involves safety and chemical hygiene training. The CEO and VPGM have the ultimate responsibility for chemical hygiene in the laboratory and provide continuing support of the program. The Chemical Hygiene Officer (CHO) has the responsibility of coordinating and enforcing the laboratory safety program at CompuChem. Each employee in the laboratory is responsible for ensuring an effective chemical hygiene program. The safety program involves the following key elements:

- safety training programs for all personnel
- the Chemical Hygiene Plan, approved by the VPGM and the CEO
- the Contingency Plan, approved by the VPGM and the CEO
- periodic (at least quarterly) inspections of the facilities for compliance with safety regulations and the safety SOPs
- verification that all safety equipment is operable and in good working condition (including inspection and recharging of all fire extinguishers, and monthly inspection of fume hood performance)
- initial testing of all new safety equipment
- periodic (at least annual) fire/evacuation drills
- the safety committee, which is comprised of safety facilitators representing key sections of the operation
- Right-to-Know seminars held for all laboratory personnel to discuss chemical hazards, safety precautions, medical treatment, and spill cleanup procedures
- monthly swipe tests

The CHO and Radiation Safety Officer (RSO) have the responsibility of conducting internal safety inspections, covering all aspects of laboratory safety including fire, hazardous materials, personal dress, electrical safety, posted evacuation routes, and condition of all safety equipment. The RSO is responsible for overseeing the safety aspects of the radiological operations in the laboratory. This includes conducting quarterly inspections and monthly swipe tests. Please refer to Section 17.3 for details on the role of the CHO/RSO and to CompuChem's *Radiological Laboratory Safety Manual* for details on safety in that laboratory. Corrective actions identified in the inspection or during safety drills are the responsibility of each laboratory section.

Yearly safety briefings for all employees are the responsibility of the department/laboratory managers. Managers receive their instruction through the CHO/RSO. Training includes safety for fire, electricity, compressed gases, chemical hazards, radiological hazards, and safety equipment,

depending on the responsibilities of the department or laboratory. New employees must be trained in all aspects of safety concerned with their job responsibilities and (when applicable) the laboratory(ies) in which they work. Human Resources, along with laboratory/department managers and the CHO/RSO must maintain documentation of safety training. The documentation must include a completed training documentation form, a list of the attendees, the training subject(s), the time spent in training, and the date.

A variety of local seminars, workshops, and lectures are also made available to employees. Again, because CompuChem is located within minutes of several university campuses and key federal agencies (U.S. Environmental Protection Agency—Environmental Research Center/Office of Air Quality Planning and Standards, National Institute of Environmental Health Sciences, Research Triangle Institute), employees have access to a variety of educational resources. Workshop and seminar attendees usually transmit their experiences in the form of trip reports or in-house presentations to appropriate staff members. CompuChem has also established an educational assistance program, reimbursing employees for the cost of formal coursework that enhances job performance and opportunities for advancement.

5.0 Analytical Procedures

CompuChem performs a variety of analytical methods. Table 5-1 lists these methods in detail and includes the published method references and applicable analytes or analyte classes. All U.S. EPA methods are performed without deviations from the published method. Any minor modifications made to other standardized methods are documented in the method summary that is included in each deliverable data package.

All analytical methods are validated before being offered as an analytical service for sale. Method validation studies are reviewed by the department manager and QA department staff, who must approve the studies. Standard operating procedures (SOPs) are then written for new methods. SOPs are also reviewed by the QA department. Any variance from standardized methods is documented in the SOP.

5.1 Limits of Detection

A formalized method detection limit (MDL) study is performed yearly for all approved methods currently in use. The studies are performed following the design specified in the Federal Register, 40 CFR Part 136 (October 26, 1984). Current MDLs for all methods can be found in Supplement E to the QA Plan. (See Section 3.0 for ordering information.)

The MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is determined from the analysis of a sample in a given matrix (using both laboratory pure water and laboratory pure sand or furnaced sodium sulfate matrices) containing the analyte. At least eight replicate samples are prepared containing the analyte(s) to be tested at a concentration that is equal to or in the same concentration range as the estimated MDL. It is recommended that the concentration fall between one and five times the estimated MDL. The samples are processed through the entire analytical method. The MDL is calculated using the standard deviation of the replicate measurements and the Student's T value at the 99% confidence level. The mean analyte value and the mean percent recovery are also calculated.

5.2 Precision and Accuracy Studies

An initial precision and accuracy demonstration is also performed for each approved method. Four replicate control samples are spiked at concentrations near the calibration midpoint and processed through the entire analytical method. The mean percent recovery and percent relative standard deviation are derived from the replicate results. Precision and accuracy data from these studies are presented in Tables 5-2 through 5-20. Statistically-derived control limits used for the evaluation of the laboratory control sample and control charting program are determined from laboratory-acquired data points and determined at two and three standard deviations. These

are used in evaluating the laboratory control sample analysis for all methods but only updated for CLP methods if actually tighter than those specified in the SOW. However, control limits for percent recovery and relative percent difference (RPD) used in routine field sample analysis are those defined in the appropriate method, and are shown in each table under the column header *Method Acceptance Limits*.

5.3 Method Validation Studies

To begin analysis of samples for written methods not currently offered, a method validation study must first be performed. The as-written method is reviewed by a chemist familiar with the extraction/preparation procedures and the instrumental detection systems required. The chemist looks for safety hazards, applicability of available instrument systems, new equipment requirements, any discrepancies in the written method, and the QA/QC requirements. A plan of testing approach to be taken is discussed with the laboratory manager and other appropriate members of senior management.

A formalized MDL study is then performed following the *Federal Register* 40 CFR Part 136, along with a precision and accuracy determination, and any other pertinent information is then forwarded to the QA department for final approval. Any deviations from the published method must be noted in the SOP. Once approval by QA and SOP formalization has occurred, analysis codes can be developed and the new method can be offered to clients. If methods are truly developed by CompuChem, more elaborate testing schemes would be required. Supplement E contains tables of the most recently determined MDLs.

Table 5-1. Analytical Methods Performed by CompuChem Environmental Corporation

Method ID	Type of Analysis	Analytes	References
Clean Water Act Methods			
160.1	manual	filterable residue total dissolved solids	EPA, March 1983
160.2	manual	non-filterable residue total suspended solids	EPA, March 1983
130.1/ 10-301-31-1-A	colorimetric, flow injection analyzer-Lachat	total hardness as $CaCO_3$	EPA, March 1983 Lachat, Nov. 1991

Table 5-1(CONTINUED). Analytical Methods Performed by CompuChem Environmental Corporation

Method ID	Type of Analysis	Analytes	References
Clean Water Act Methods (continued)			
200.7	inductively coupled plasma (ICP)	inorganic metals	40 CFR 136, Appendix C
206.2	graphite furnace AA (GFAA)	arsenic	EPA, March 1983
239.2	GFAA	lead	EPA, March 1983
245.1/245.5	cold vapor AA (CVAA)	mercury	EPA, March 1983
270.2	GFAA	selenium	EPA, March 1983
279.2	GFAA	thallium	EPA, March 1983
310.2, 10-303-31-1-A	colorimetric, flow injection analyzer-Lachat	alkalinity	EPA, March 1983 Lachat, Dec.1988
325.2, 10-117-07-1-A	colorimetric, flow injection analyzer-Lachat	chloride	EPA, March 1983 Lachat, Oct.1991
335.2/.3	manual distillation, automated Technicon	cyanide	EPA, March 1983
350.1, 10-107-06-1-A	colorimetric, flow injection analyzer-Lachat	ammonia	EPA, March 1983 Lachat, Sept.1991
375.4	turbidimetric	sulfate	EPA, March 1983
420.1/.2	manual distillation, automated Technicon	phenols aqueous (A)	EPA, March 1983

Table 5-1 (CONTINUED). Analytical Methods Performed by CompuChem Environmental Corporation

Method ID	Type of Analysis	Analytes	References
Clean Water Act Methods (continued)			
505	DC-180 total organic carbon (TOC) analyzer	TOC	EPA, December 1983 Standard Methods, 16th Edition, 1985
506	DX-208 total organic halides (TOX) analyzer	TOX	EPA, December 1983 Standard Methods, 16th Edition, 1985
601	GC purge and trap (P&T), Hall detector	purgeable halocarbons	40 CFR 136, Appendix A
602	GC P&T PID detector	purgeable aromatics	40 CFR 136, Appendix A
608	extraction, GC/ECD	organochlorine pesticides & PCBs	40 CFR 136, Appendix A
610	HPLC, UV & fluorescence detectors in series	PAHs (A)	40 CFR 136, Appendix A
624	GC/MS P&T Megabore column	purgeable volatile organics	40 CFR 136, Appendix A
625	extraction, GC/MS capillary column	acid and base/neutral (B/N) extractables	40 CFR 136, Appendix A
340.2, 10-109-12-2-A	ion selective electrode, flow injection analyzer-Lachat	total fluorides (without distillation)	EPA, March 1983 Lachat, 1989
353.2	colorimetric, automated with cadmium reduction	nitrate	EPA, March 1983

Table 5-1 (CONTINUED). Analytical Methods Performed by CompuChem Environmental Corporation

Method ID	Type of Analysis	Analytes	References
Clean Water Act Methods (continued)			
353.2	colorimetric, automated without cadmium reduction	nitrite	EPA, March 1983
503B	Fourier transform infrared (FT-IR)	oil and grease (A)	Standard Methods, 16th Edition, 1985
503 E	FT-IR	total petroleum (A) hydrocarbons (TPH)	Standard Methods, 16th Edition, 1985
418.1	FT-IR	TPH (S) O&G (S)	EPA, March 1983
10-124-13-1-A	colorimetric, flow injection analyzer-Lachat	hexavalent chromium Cr+	Lachat 1991
504	microextraction, GC/ECD	EDB and DBCP	EMSL 6/85, 11/85
Resource Conservation and Recovery Act Methods			
1311	TCLP	semivolatiles, pest., herb., metals	40 CFR 261 Appendix II, EPA SW-846 3rd. Edition
1311	TCLP with zero headspace extraction	volatile organics	40 CFR 261, Appendix II, EPA SW-846 3rd. Edition
3005	aqueous acid digestion	total recoverable or dissolved metals	EPA SW-846, 3rd. Edition
3010	aqueous acid digestion	total metals	EPA SW-846 3rd Edition
3020	aqueous acid digestion	total metals	EPA SW-846 3rd. Edition

Table 5-1 (CONTINUED). Analytical Methods Performed by CompuChem Environmental Corporation

Method ID	Type of Analysis	Analytes	References
Resource Conservation and Recovery Act Methods (continued)			
3050	soil/sediment/ sludge acid digestion	total metals	EPA SW-846 3rd. Edition
3510	separatory funnel, liquid-liquid extraction	organic extractables (A)	EPA SW-846 3rd. Edition
3520	continuous liquid-liquid extraction	organic extractables (A)	EPA SW-846 3rd. Edition
3540	Soxhlet extraction	extractable organics (S)	EPA SW-846 3rd. Edition
3550	sonication extraction	nonvolatile and extractable organics	EPA SW-846 3rd. Edition
6010	ICP	inorganic metals	EPA SW-846 3rd. Edition
7060	GFAA	arsenic	EPA SW-846 3rd. Edition
7195/7191	GFAA	hexavalent chromium	EPA SW-846 3rd. Edition
7421	GFAA	lead	EPA SW-846 3rd. Edition
7470/7471	manual CVAA	mercury	EPA SW-846 3rd. Edition
7740	GFAA	selenium	EPA SW-846 3rd. Edition

Table 5-1 (CONTINUED). Analytical Methods Performed by CompuChem Environmental Corporation

Method ID	Type of Analysis	Analytes	References
Resource Conservation and Recovery Act Methods (continued)			
7841	GFAA	thallium	EPA SW-846 3rd. Edition
8010	GC/Hall detector	halogenated volatile organics	EPA SW-846 3rd. Edition
8015 (modified)	GC/FID detector	jet fuel/gasoline/ diesel	EPA SW-846 3rd. Edition
8020	GC/PID detector	aromatic volatile organics	EPA SW-846 3rd. Edition
Chapter 7 9010	reflux distillation automated Technicon	cyanide, reactive sulfide, reactive	EPA SW-846 3rd. Edition
9010	manual distillation automated Technicon	cyanide, total and amenable	EPA SW-846 3rd. Edition
Chapter 7, 9030	acid distillation, titration	sulfide, reactive	EPA SW-846 3rd. Edition
8080	GC/ECD detector	organochlorine pesticides and PCBs	EPA SW-846 3rd. Edition
3620	Florisil column cleanup	organochlorine pesticides & PCBs	EPA SW-846 3rd. Edition
3640	gel permeation cleanup	organochlorine pesticides & PCBs semivolatile organic extractables	EPA SW-846 3rd. Edition
8140	GC/flame nitrogen phosphorus detector (NPD)	organophosphorus pesticides	EPA SW-846 3rd. Edition

Table 5-1 (CONTINUED). Analytical Methods Performed by CompuChem Environmental Corporation

Method ID	Type of Analysis	Analytes	References
Resource Conservation and Recovery Act Methods (continued)			
8150	GC/ECD	chlorinated herbicides	EPA SW-846 3rd. Edition
8240	GC/MS P&T	purgeable volatile organics	EPA SW-846 3rd. Edition
8270	GC/MS capillary column	semivolatile organic extractables	EPA SW-846 3rd. Edition
8310	HPLC-UV and fluorescence detectors in series	polynuclear aromatic hydrocarbons (PAHs)	EPA SW-846 3rd. Edition
3630	silica gel cleanup	PAHs	EPA SW-846 3rd. Edition
9012	manual distillation, automated Technicon	cyanide	EPA SW-846 3rd. Edition
9038	turbidimetric	sulfate	EPA SW-846 3rd. Edition & updates
9020	DX-208 TOX analyzer	TOX	EPA SW-846 3rd. Edition
9071/418.1	Soxhlet extraction FT-IR	O&G solid (S)	EPA SW-846 3rd. Edition\ EPA March 1983
9251 10-117-07-1-A	colorimetric, flow injection analyzer-Lachat	chloride	EPA SW-846 3rd. Edition
9065/9066	manual distillation, automated Technicon	phenol	EPA SW-846 3rd. Edition

Table 5-1 (CONTINUED). Analytical Methods Performed by CompuChem Environmental Corporation

Method ID	Type of Analysis	Analytes	References
Superfund/Comprehensive Environmental Response, Compensation, and Liability Act			
EPA Contract Laboratory Program (CLP)	GC/MS P&T Megabore column	purgeable volatile organics	CLP Statement-of-Work (SOW) 3/90, OLM01.9
EPA CLP	extraction, GC/MS capillary column	semivolatile organic extractables	CLP SOW 3/90, OLM01.9
EPA CLP	extraction, GC/ECD detector	organochlorine pesticides & PCBs	CLP SOW 3/90, OLM01.9
EPA CLP	ICP (modified 200.7)	inorganic metals	CLP SOW 3/90 ILM03.0
EPA CLP	GFAA	inorganic metals (arsenic, selenium, lead, thallium)	CLP SOW 3/90 ILM03.0
EPA CLP	CVAA	mercury	CLP SOW 3/90, ILM03.0
EPA CLP	manual distillation, automated Technicon	cyanides	CLP SOW 3/90 ILMO3.0
EPA CLP	* ICP, GFAA, CVAA	dissolved metals (including mercury)	EPA CLP 3/90 ILMO3.0
EPA Superfund Analytical Methods (SAM)	GC/MS	low concentration purgeable volatile organics	SAM 10/92
EPA SAM	GC/MS	low concentration SV organic extractables	SAM 10/92

*It is our policy not to digest samples for dissolved metals requiring CLP analysis. Since HAZWRAP samples require a digestion, on a project-specific basis, samples will be digested as required.

Table 5-1 (CONTINUED). Analytical Methods Performed by CompuChem Environmental Corporation

Method ID	Type of Analysis	Analytes	References
EPA SAM	GC	low concentration organo-chlorine pesticide/PCBs	SAM 10/92
EPA	GC P&T, PID/ELCD	purgeable volatile organics	draft SOW for QTM 2/93
EPA	solid phase/solvent extraction, GC/ECD GC/FID GC/PID or FID	pesticides, PCBs as arochlors, polynuclear arom. hydrocarb. phenolic compounds	CLP draft SOW for QTM 2/93 (PAHs)
Radiological Parameters			
EPA 9310	gas proportional counter	gross alpha	EPA SW-846 3rd. Edition
EPA 9310	gas proportional counter	gross beta	EPA SW-846 3rd. Edition
EPA 9315	gas proportional counter	total radium	EPA SW-846 3rd. Edition
EPA 903.1 (modified)	gas scintillation counter	radium-226	EPA, August 1980
U-02 (modified)	silicon surface barrier detector, alpha spectrometer	isotopic uranium-234, 238	HASL-300

Method Reference Key

40 CFR 136, Appendix A
40 CFR 136, Appendix C

Code of Federal Regulations 40, Part 136. (1984). *Test procedures for analysis of organic pollutants, part VIII Environmental Protection Agency 40 CFR Part 136 guidelines establishing test procedures for the analysis of pollutants under the Clean Water Act's final rule and interim final rule and proposed rule.*
Appendix A. (October 26, 1984). *Methods for organic chemical analysis of municipal and industrial wastewater.*
Appendix A. (October 26, 1984). *Inductively coupled plasma-atomic emission spectrometric method for trace element analysis of water and wastes, method 200.7.*

Method Reference Key

CLP SOW 3/90	U.S. EPA. (March 1990). *Contract Laboratory Program Statement-of-Work for organic and inorganic analysis, multi-media, multi-concentration.* Document number OLM01.0 with revisions through OLM01.9 (organic), and number ILMO3.0 (inorganic).
EPA QTM 2/93	U.S. EPA. (February 1993). draft *Contract Laboratory Program Statement-of-Work for quick turnaround method (QTM).*
EPA 10/92 SAM	U.S. EPA. (October 1992). *U.S. EPA Superfund analytical methods for low concentration organics in water.*
40 CFR 261	U.S. EPA. (November 24, 1992). *Toxicity characteristic leaching procedure (TCLP).* Appendix I added by 57 CFR 55114. Toxicity Characteristics revision, Final Rule.
Lachat 1989/1991	Lachat Instruments. (1989). *Method manual for the QuikChem automated ion analyzer.* Milwaukee, WI.
EPA August 1980	U.S. EPA. (August 1980). *Prescribed procedures for measurement of radioactivity in drinking water.* EPA-600/4-80-032.
HASL-300	U.S. DOE Environmental Measurements Laboratory. (November 1990). *HASL-300,* 27th Edition.
EMSL-Cincinnati 11/85	Environmental Monitoring Systems Laboratory. (November 1985). *Methods for the determination in finished drinking water and raw source water,* 6/85 (revised 11/85), EMSL Physical/Chemical Methods Branch.
Standard Methods 16th Edition	APHA, AWWA, WPCF. (1985). *Standard methods for the examination of water and wastewater.* 16th. Edition.

Tables 5-2 through 5-34 contain precision, accuracy, and spiking level information for the analyses performed by CompuChem. Precision and accuracy information for methods not covered in these tables will be added in subsequent updates to this QA Plan.

Table 5-2. Precision and Accuracy for U.S. EPA CLP SOW 3/90 Semivolatile Aqueous Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
phenol	4.4	76-99	87	12-110	42
bis(2-chloroethyl) ether	4.2	77-100	88		
2-chlorophenol	4.8	74-99	87	27-123	40
1,3-dichlorobenzene	4.0	71-91	81		
1,4-dichlorobenzene	4.4	70-92	81	36-97	28
1,2-dichlorobenzene	6.2	61-89	75		
2-methylphenol	5.2	67-91	79		
2,2'-oxybis(1-chloropropane)	3.9	77-98	87		
4-methylphenol	4.7	61-81	71		
N-nitroso-Di-N-propylamine	3.9	73-92	83	41-116	38
hexachloroethane	4.4	68-88	78		
nitrobenzene	4.1	84-108	96		
isophorone	3.3	83-101	92		
2-nitrophenol	4.4	95-124	110		
2,4-dimethylphenol	9.7	77-123	100		
bis(2-chloroethoxy) methane	4.3	82-106	94		
2,4-dichlorophenol	3.3	85-103	94		
1,2,4-trichlorobenzene	3.5	90-111	100	39-98	28
naphthalene	4.2	85-109	97		
4-chloroaniline	4.1	83-106	94		
hexachlorobutadiene	3.7	87-109	98		
4-chloro-3-methylphenol	4.4	85-111	98	23-97	42
2-methylnaphthalene	3.3	67-82	74		
hexachlorocyclopentadiene	11	50-86	11		
2,4,6-trichlorophenol	5.6	82-116	99		
2,4,5-trichlorophenol	2.8	90-107	98		
2-chloronaphthalene	3.2	76-91	84		
2-nitroaniline	3.4	82-101	92		
dimethyl phthalate	3.6	79-99	89		
acenaphthylene	3.6	69-86	77		
3-nitroaniline	4.7	86-117	102		
acenaphthene	3.1	72-87	80	46-118	31
2,4-dinitrophenol	4.3	50-62	56		
4-nitrophenol	6.2	91-132	111	10-80	50
dibenzofuran	4.0	73-94	84		
2,4-dinitrotoluene	3.5	80-100	90	24-96	38
2,6-dinitrotoluene	3.4	79-97	88		
diethyl phthalate	4.0	74-93	83		

Table 5-2 (CONTINUED). Precision and Accuracy for U.S. EPA CLP SOW 3/90 Semivolatile Aqueous Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
4-chlorophenyl phenyl ether	3.5	68-84	76		
fluorene	3.5	72-90	81		
4-nitroaniline	4.4	98-127	112		
4,6-dinitro-2-methylphenol	12	67-121	94		
N-nitrosodiphenylamine	3.3	76-97	84		
4-bromophenyl phenyl ether	2.9	93-110	102		
hexachlorobenzene	3.6	82-102	92		
pentachlorophenol	10	65-104	84	9-103	50
phenanthrene	3.3	98-119	108		
carbazole	3.3	85-104	95		
anthracene	4.2	85-110	98		
di-N-butyl phthalate	3.9	78-99	88		
fluoranthene	2.9	95-113	104		
pyrene	1.8	86-96	91	26-127	31
butylbenzyl phthalate	4.1	69-88	78		
3,3'-dichlorobenzidine	7.9	45-73	59		
benzo(a)anthracene	3.2	86-104	95		
bis(2-ethylhexyl) phthalate	6.2	65-95	80		
chrysene	4.3	64-83	73		
di-N-octyl phthalate	3.9	84-106	95		
benzo(b) fluoranthene	4.6	83-110	97		
benzo(k) fluoranthene	4.8	41-55	48		
benzo(a) pyrene	3.7	72-90	81		
indeno (1,2,3-c,d) pyrene	3.6	71-88	80		
dibenzo (a,h) anthracene	5.7	69-98	84		
benzo (g,h,i) peryiene	5.4	58-80	69		
Surrogates					
2-fluorophenol	4.1	82-105	93	21-110	N/A
D5-phenol	4.6	77-101	89	10-110	N/A
2-chlorophenol-D4	4.2	52-67	59	33-110	N/A
1,2-dichlorobenzene-D4	6.0	57-83	70	16-110	N/A
D5-nitrobenzene	3.8	87-109	98	35-114	N/A
2-fluorobiphenyl	2.9	79-94	87	43-116	N/A
2,4,6-tribromophenol	4.5	77-101	89	10-123	N/A
D14-terphenyl	1.3	107-116	111	33-141	N/A*

*Not applicable (N/A)

Table 5-3. Precision and Accuracy for U.S. EPA CLP SOW 3/90 Low Level Semivolatile Solids

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria %R Limits	%RPD
phenol	11	43-86	65	26-90	35
bis(2-chloroethyl) ether	11	45-87	66		
2-chlorophenol	11	41-84	63	25-102	50
1,3-dichlorobenzene	10	43-82	62		
1,4-dichlorobenzene	11	43-84	63	28-104	27
1,2-dichlorobenzene	11	42-82	62		
2-methylphenol	9.3	43-77	60		
2,2'-oxybis(1-chloropropane)	12	47-96	72		
4-methylphenol	9.8	46-84	65		
N-nitroso-Di-N-propylamine	11	46-92	69	41-126	38
hexachloroethane	10	45-86	70		
nitrobenzene	7.9	53-85	69		
isophorone	5.5	59-82	70		
2-nitrophenol	8.5	52-89	70		
2,4-dimethylphenol	5.9	27-38	32		
bis(2-chloroethoxy) methane	6.6	55-83	69		
2,4-dichlorophenol	6.6	52-78	65		
1,2,4-trichlorobenzene	8.8	48-82	65	38-107	23
naphthalene	6.6	50-75	63		
4-chloroaniline	6.9	25-39	32		
hexachlorobutadiene	8.4	49-82	65		
4-chloro-3-methylphenol	7.0	58-89	74	26-103	33
2-methylnaphthalene	6.6	44-65	55		
hexachlorocyclopentadiene	6.7	50-75	62		
2,4,6-trichlorophenol	38	D-210	98		
2,4,5-trichlorophenol	37	D-209	99		
2-chloronaphthalene	5.3	51-71	61		
2-nitroaniline	4.6	71-94	83		
dimethyl phthalate	6.2	56-82	69		
acenaphthylene	5.0	50-68	59		
3-nitroaniline	9.3	27-48	37		
acenaphthene	5.4	54-74	64	31-137	19
2,4-dinitrophenol	12	64-74	69		
4-nitrophenol	4.7	72-96	84	11-114	50
dibenzofuran	4.8	54-72	63		
2,4-dinitrotoluene	5.1	63-86	74	28-89	47
2,6-dinitrotoluene	6.7	56-84	70		
diethyl phthalate	5.7	57-81	69		

Table 5-3 (CONTINUED). Precision and Accuracy for U.S. EPA CLP SOW 3/90 Low Level Semivolatile Solids

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria %R Limits	%RPD
4-chlorophenyl phenyl ether	6.1	53-77	65		
fluorene	5.9	52-75	63		
4-nitroaniline	7.1	58-90	74		
4,6-dinitro-2-methylphenol	7.7	59-94	77		
N-nitrosodiphenylamine	6.6	44-66	55		
4-bromophenyl phenyl ether	7.4	51-81	66		
hexachlorobenzene	8.6	46-79	63		
pentachlorophenol	9.4	49-88	69	17-109	47
phenanthrene	6.5	54-81	68		
carbazole	5.4	57-79	68		
anthracene	6.4	52-76	64		
di-N-butyl phthalate	6.6	52-78	65		
fluoranthene	6.1	54-78	66		
pyrene	8.3	48-80	64	35-142	36
butylbenzyl phthalate	6.1	52-76	64		
3,3'-dichlorobenzidine	20	3-10	6		
benzo(a)anthracene	8.4	53-89	71		
bis(2-ethylhexyl) phthalate	7.0	57-85	68		
chrysene	8.5	45-76	60		
di-N-octyl phthalate	7.6	55-87	71		
benzo(b) fluoranthene	7.3	57-89	73		
benzo(k) fluoranthene	10	30-56	43		
benzo(a) pyrene	7.8	50-81	66		
indeno (1,2,3-c,d) pyrene	10	60-112	86		
dibenzo (a,h) anthracene	7.9	62-100	81		
benzo (g,h,i) perylene	15	40-108	74		
Surrogates:					
nitrobenzene-D5	8.3	85-142	114	23-120	N/A
2-fluorobiphenyl	4.9	84-113	99	30-115	N/A
terphenyl-D14	7.4	93-147	120	18-137	N/A
phenol-D5	10	49-93	71	24-113	N/A
2-fluorophenol	11	49-95	72	25-121	N/A
2,4,6-tribromophenol	6.0	66-95	81	19-122	N/A
2-chlorophenol-D4	9.8	35-64	50	20-130	N/A
1,2-dichlorobenzene-D4	11	67-134	101	20-130	N/A

Table 5-4. Precision and Accuracy for U.S. EPA CLP SOW 3/90 Medium Level Semivolatile Solids

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
phenol	5.5	72-101	87	26-90	35
bis(2-chloroethyl) ether	7.1	71-110	91		
2-chlorophenol	6.6	68-101	84	25-102	50
1,3-dichlorobenzene	6.3	69-101	85		
1,4-dichlorobenzene	6.6	67-100	83	28-104	27
1,2-dichlorobenzene	7.7	65-104	85		
2-methylphenol	8.5	44-73	59		
2,2'-oxybis(1-chloropropane)	7.7	69-110	89		
4-methylphenol	8.1	47-80	63		
N-nitroso-Di-N-propylamine	6.7	69-103	86	41-126	38
hexachloroethane	6.8	65-99	82		
nitrobenzene	5.6	76-106	91		
isophorone	6.0	81-116	98		
2-nitrophenol	6.4	81-120	100		
2,4-dimethylphenol	12	25-55	40		
bis(2-chloroethoxy) methane	6.3	76-112	94		
2,4-dichlorophenol	6.1	70-102	86		
1,2,4-trichlorobenzene	6.5	73-108	91	38-107	23
naphthalene	6.4	74-109	92		
4-chloroaniline	8.7	28-48	38		
hexachlorobutadiene	6.9	66-101	84		
4-chloro-3-methylphenol	2.8	72-86	79	26-103	33
2-methylnaphthalene	7.2	59-92	75		
hexachlorocyclopentadiene	4.6	55-72	63		
2,4,6-trichlorophenol	5.2	79-108	94		
2,4,5-trichlorophenol	3.9	80-100	90		
2-chloronaphthalene	4.7	78-103	91		
2-nitroaniline	4.9	81-109	95		
dimethyl phthalate	5.4	77-107	92		
acenaphthylene	4.5	70-93	81	31-137	19
3-nitroaniline	9.5	45-80	63		
acenaphthene	5.2	80-110	95		
2,4-dinitrophenol	8.7	77-131	104		
4-nitrophenol	8.6	76-130	103	11-114	50
dibenzofuran	5.1	75-102	89		
2,4-dinitrotoluene	6.9	76-115	96	28-89	47
2,6-dinitrotoluene	4.9	77-104	90		
diethyl phthalate	5.5	78-108	93		

Table 5-4 (CONTINUED). Precision and Accuracy for U.S. EPA CLP SOW 3/90 Medium Level Semivolatile Solids

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
4-chlorophenyl phenyl ether	5.3	80-110	95		
fluorene	5.5	78-108	93		
4-nitroaniline	6.6	40-59	49		
4,6-dinitro-2-methylphenol	5.0	91-123	107		
N-nitrosodiphenylamine	4.9	53-71	62		
4-bromophenyl phenyl ether	3.6	83-103	93		
hexachlorobenzene	3.4	77-94	85		
pentachlorophenol	4.3	89-115	102	17-109	47
phenanthrene	4.3	82-107	95		
carbazole	5.5	66-93	80		
anthracene	3.3	76-92	84		
di-N-butyl phthalate	4.3	78-102	90		
fluoranthene	4.7	78-103	90		
pyrene	5.2	65-90	77	35-142	36
butylbenzyl phthalate	3.3	72-87	80		
3,3'-dichlorobenzidine	13	29-65	47		
benzo(a)anthracene	5.8	76-109	92		
bis(2-ethylhexyl) phthalate	2.5	81-94	88		
chrysene	10	46-85	65		
di-N-octyl phthalate	4.7	77-102	89		
benzo(b) fluoranthene	6.3	75-110	92		
benzo(k) fluoranthene	9.7	34-62	48		
benzo(a) pyrene	4.4	68-88	78		
indeno (1,2,3-c,d) pyrene	5.0	85-115	100		
dibenzo (a,h) anthracene	3.7	93-115	104		
benzo (g,h,i) perylene	2.2	68-77	72		
Surrogates:					
2-fluorophenol	3.7	78-97	88	25-121	N/A
D5-phenol	5.7	75-105	90	24-113	N/A
2-chlorophenol-D4	5.9	51-74	62	20-130	N/A
1,2-dichlorobenzene-D4	7.1	69-105	87	20-130	N/A
D5-nitrobenzene	5.7	83-117	100	23-120	N/A
2-fluorobiphenyl	4.2	82-106	94	30-115	N/A
2,4,6-tribromophenol	6.0	74-106	90	19-122	N/A
D14-terphenyl	5.7	77-108	93	18-137	N/A

Table 5-5. Precision and Accuracy for U.S. EPA CLP SOW 3/90 Volatile Aqueous GC/MS Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
chloromethane	24	13.3-82	48		
vinyl chloride	14	38-94	67		
bromomethane	11	54-104	79		
chloroethane	8.5	62-105	83		
1,1-dichloroethene	6.0	72-103	88	61-145	14
acetone	5.5	82-114	98		
carbon disulfide	2.1	86-98	92		
methylene chloride	3.6	84-105	94		
trans-1,2-dichloroethene	3.1	81-97	89		
1,1-dichloroethane	2.6	82-96	89		
cis-1,2-dichloroethene	5.1	78-106	92		
2-butanone	13	78-183	131		
chloroform	2.8	80-95	87		
1,1,1-trichloroethane	2.7	91-107	99		
carbon tetrachloride	2.4	88-102	95		
benzene	1.9	90-101	95	76-127	11
1,2-dichloroethane	2.2	89-102	95		
trichloroethene	3.0	87-104	95	71-120	14
1,2-dichloropropane	1.2	95-102	98		
bromodichloromethane	1.4	94-102	98		
4-methyl-2-pentanone	4.7	96-127	112		
toluene	2.2	85-98	91	76-125	13
cis-1,3-dichloropropene	2.1	89-100	94		
trans-1,3-dichloropropene	1.3	94-102	98		
1,1,2-trichloroethane	1.1	94-100	97		
tetrachloroethene	2.1	87-99	93		
2-hexanone	6.3	89-131	110		
dibromochloromethane	.72	94-99	97		
chlorobenzene	2.8	81-96	88	75-130	13
ethylbenzene	2.5	86-99	93		
m,p-xylenes	2.2	86-99	93		
o-xylene	3.0	81-97	89		
styrene	1.7	84-94	89		
bromoform	1.9	97-109	103		
1,1,2,2-tetrachloroethane	4.2	88-114	101		
total 1,2-dichloroethene	3.9	80-101	91		
total xylenes	2.5	85-98	91		
Surrogates:					
toluene-D8	3.3	95-105	100	88-110	NA
bromofluorobenzene	2.5	98-106	102	86-115	NA
1,2-dichloroethane-D4	10	88-120	104	76-114	NA

Table 5-6. Precision and Accuracy for U.S. EPA CLP SOW 3/90 Low Level Volatiles Solid GC/MS Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
chloromethane	7.6	85-136	111		
vinyl chloride	2.0	109-115	112		
bromomethane	20	52-203	128		
chloroethane	9.8	94-173	134		
1,1-dichloroethene	3.6	105-131	118	59-172	22
carbon disulfide	4.6	101-133	117		
acetone	47	D-127	53		
methylene chloride	5.4	96-133	114		
trans-1,2-dichloroethene	3.5	102-126	114		
1,1-dichloroethane	3.5	105-129	117		
cis-1,2-dichloroethene	2.6	113-132	122		
2-butanone	15	39-103	71		
chloroform	3.5	105-129	117		
1,1,1-trichloroethane	4.1	86-110	98		
carbon tetrachloride	3.1	86-103	94		
benzene	13	49-113	81	66-142	21
1,2-dichloroethane	1.4	105-115	110		
trichloroethene	3.3	91-111	101	62-137	24
1,2-dichloropropane	1.9	95-107	101		
bromodichloromethane	1.8	98-109	103		
cis-1,3-dichloropropene	2.0	99-111	105		
4-methyl-2-pentanone	11	52-101	77		
toluene	3.1	93-111	102	59-139	21
trans-1,3-dichloropropene	1.2	101-109	105		
1,1,2-trichloroethane	3.3	87-106	97		
tetrachloroethene	3.0	94-113	104		
2-hexanone	8.6	56-95	76		
dibromochloromethane	2.8	91-108	99		
chlorobenzene	3.0	93-112	102	60-133	21
ethylbenzene	2.3	97-111	104		
m,p-xylenes	2.0	90-101	96		
o-xylene	2.1	94-107	101		
styrene	1.35	97-105	101		
bromoform	4.4	84-110	97		
1,1,2,2-tetrachloroethane	3.0	90-107	98		
Surrogates:					
D4-1,2-dichloroethane	0.93	97-103	100	70-121	N/A
bromofluorobenzene	3.4	83-102	92	59-113	N/A
D8-toluene	4.9	77-104	91	84-138	N/A

Table 5-7. Precision and Accuracy for U.S. EPA CLP SOW 3/90 Medium Level Volatiles Solid GC/MS Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
chloromethane	43	D-246	116		
vinyl chloride	29	13-175	94		
bromomethane	3.5	119-147	133		
chloroethane	3.1	123-148	135		
1,1-dichloroethene	8.7	68-116	92	59-172	22
acetone	3.5	73-90	81		
carbon disulfide	5.5	88-122	105		
methylene chloride	7.2	69-107	88		
trans-1,2-dichloroethene	1.7	91-101	96		
1,1-dichloroethane	1.7	94-104	99		
cis-1,2-dichloroethene	1.3	87-95	91		
2-butanone	5.7	86-121	103		
chloroform	1.2	90-97	94		
1,1,1-trichloroethane	.75	94-98	96		
carbon tetrachloride	.75	96-101	99		
benzene	.86	100-105	102	66-142	21
1,2-dichloroethane	1.3	86-94	90		
trichloroethene	1.7	92-98	95	62-137	24
1,2-dichloropropane	6.4	71-102	88		
bromodichloromethane	2.8	75-89	82		
4-methyl-2-pentanone	6.8	87-131	109		
toluene	1.9	98-110	104	59-139	21
cis-1,3-dichloropropene	2.5	107-124	116		
trans-1,3-dichloropropene	3.9	109-138	123		
1,1,2-trichloroethane	3.6	96-119	107		
tetrachloroethene	2.6	84-98	91		
2-hexanone	6.4	96-141	119		
dibromochloromethane	3.2	99-119	109		
chlorobenzene	2.0	91-102	96	60-133	21
ethylbenzene	1.5	96-106	101		
m,p-xylenes	1.4	97-105	101		
o-xylene	1.3	96-104	100		
styrene	1.7	91-101	96		
bromoform	6.9	88-133	111		
1,1,2,2-tetrachloroethane	5.5	81-113	97		
total 1,2-dichloroethene	1.4	90-97	93		
total xylenes	1.3	97-105	101		
Surrogates:					
D4-1,2-dichloroethane	1.0	91-97	94	70-121	N/A
bromofluorobenzene	1.3	99-107	103	59-113	N/A
toluene-D8	1.3	102-111	106	84-138	N/A

Table 5-8. Precision and Accuracy for Method 8240 Medium Level Volatiles Solid GC/MS Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
chloromethane	8.9	67-116	92		
vinyl chloride	8.3	65-107	86		
bromomethane	6.6	67-100	83		
chloroethane	9.0	64-111	88		
trichlorofluoromethane	4.5	67-88	77		
acrolein	9.4	53-95	74		
1,1-dichloroethene	4.7	77-102	90	59-172	22
carbon disulfide	7.0	26-40	33		
iodomethane	6.4	85-96	80		
1,1,1-trichloro-2,2,2-trifluoroethane	7.2	70-109	89		
1,1,2-trichloro-1,2,2-trifluoroethane	6.7	71-107	89		
acetone	7.0	63-96	80		
3-chloropropene	6.2	85-124	104		
methylene chloride	1.9	65-72	68		
trans-1,2-dichloroethene	3.1	84-101	93		
acrylonitrile	5.6	66-93	80		
1,1-dichloroethane	4.2	85-109	97		
vinyl acetate	5.6	72-101	87		
cis-1,2-dichloroethene	2.3	105-121	113		
2-butanone	7.5	71-113	92		
chloroform	2.4	90-105	98		
1,1,1-trichloroethane	3.1	94-113	103		
carbon tetrachloride	3.9	90-114	102		
benzene	3.6	85-106	96	66-142	21
1,2-dichloroethane	4.3	78-102	90		
crotonaldehyde	16	63-172	117		
trichloroethene	3.4	84-103	93	62-137	24
dibromomethane	4.3	67-86	77		
1,2-dichloropropane	3.6	86-106	96		
bromodichloromethane	3.3	90-110	100		
2-chloroethyl vinyl ether	37	D-339	160		
cis-1,3-dichloropropene	2.9	93-110	102		
4-methyl-2-pentanone	3.8	67-85	76		
toluene	2.5	93-108	101	59-139	21
trans-1,3-dichloropropene	3.1	89-107	98		
1,1,2-trichloroethane	4.1	78-100	89		
ethylmethacrylate	1.6	82-90	86		
tetrachloroethene	3.2	88-106	97		
2-hexanone	4.1	67-86	76		
dibromochloromethane	3.9	80-101	90		
1,2-dibromoethane	3.8	71-90	81		
chlorobenzene	2.7	89-105	97	60-133	21

Table 5-8 (CONTINUED). Precision and Accuracy for Method 8240 Medium Level Volatiles Solid Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
1,1,1,2-tetrachloroethane	2.4	86-99	93		
ethylbenzene	2.8	87-103	95		
m,p-xylenes	2.0	81-91	86		
o-xylene	2.6	88-103	95		
styrene	2.2	88-100	94		
bromoform	3.8	68-86	77		
cis-1,4-dichloro-2-butene	4.2	68-88	78		
1,2,3-trichloropropane	3.3	77-94	86		
1,1,2,2-tetrachloroethane	2.9	78-93	86		
trans-1,4-dichloro-2-butene	3.4	79-97	88		
1,2-dibromo-3-chloropropane	5.3	65-89	77		
Surrogates:					
D4-1,2-dichloroethane	2.6	90-105	97	70-121	N/A
bromofluorobenzene	2.5	92-107	100	74-121	N/A
D8-toluene	3.7	97-121	109	81-117	N/A

Table 5-9. Precision and Accuracy for Method 8240 Low Level Volatiles Solid Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
chloromethane	2.6	135-158	147		
vinyl chloride	5.5	104-145	125		
bromomethane	2.6	106-123	147		
chloroethane	12	80-174	127		
trichlorofluoromethane	13	77-171	124		
acrolein	17	69-214	142		
1,1-dichloroethene	6.8	83-125	104	59-172	22
carbon disulfide	5.8	106-151	128		
iodomethane	15	40-108	74		
1,1,1-trichloro-2,2,2-trifluoroethane	11	63-123	93		
1,1,2-trichloro-1,2,2-trifluoroethane	5.2	83-133	98		
acetone	14	52-131	92		
3-chloropropene	6.5	93-139	116		
methylene chloride	5.6	74-104	89		
trans-1,2-dichloroethene	7.9	83-134	108		
acrylonitrile	8.3	95-157	126		
1,1-dichloroethane	4.3	108-139	123		
vinyl acetate	9.3	90-159	125		
cis-1,2-dichloroethene	4.6	97-128	112		
2-butanone	7.9	84-135	109		
chloroform	6.3	92-135	113		
1,1,1-trichloroethane	6.5	87-129	108		
carbon tetrachloride	5.4	90-124	107		
benzene	5.3	93-128	111	66-142	21
1,2-dichloroethane	4.9	94-127	110		
crotonaldehyde	13	59-135	97		
trichloroethene	5.4	81-112	97	62-137	24
dibromomethane	7.3	86-134	110		
1,2-dichloropropane	3.9	78-98	88		
bromodichloromethane	6.7	86-130	108		
2-chloroethyl vinyl ether	9.8	96-175	135		
cis-1,3-dichloropropene	6.8	87-132	109		
4-methyl-2-pentanone	8.1	84-138	111		
toluene	5.9	92-132	112	59-139	21
trans-1,3-dichloropropene	4.9	102-137	120		
1,1,2-trichloroethane	6.0	79-113	96		
ethylmethacrylate	6.2	98-143	121		
tetrachloroethene	4.7	82-109	96		
2-hexanone	13	81-183	132		
dibromochloromethane	4.9	80-108	94		
1,2-dibromoethane	5.3	78-107	93		

Table 5-9 (CONTINUED). Precision and Accuracy for Method 8240 Low Level Volatiles Solid Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
chlorobenzene	4.8	88-118	103	60-133	21
1,1,1,2-tetrachloroethane	4.5	77-101	89		
ethylbenzene	4.2	88-114	101		
m,p-xylenes	2.6	96-113	104		
o-xylene	3.3	94-115	104		
styrene	2.7	95-111	103		
bromoform	4.1	80-103	91		
cis-1,4-dichloro-2-butene	6.0	80-115	97		
1,2,3-trichloropropane	5.2	80-110	95		
1,1,2,2-tetrachloroethane	5.7	91-128	109		
trans-1,4-dichloro-2-butene	6.5	106-158	132		
1,2-dibromo-3-chloropropane	6.8	58-138	98		
Surrogates:					
D4-1,2-dichloroethane	3.3	109-121	115	70-121	N/A
bromofluorobenzene	0.91	110-113	111	74-121	N/A
D8-toluene	1.7	107-111	109	81-117	N/A

Table 5-10. Precision and Accuracy for Method 8240 Aqueous Volatiles Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
chloromethane	7.3	84-132	108		
vinyl chloride	6.8	84-128	106		
bromomethane	3.7	92-115	104		
chloroethane	5.4	86-120	103		
trichlorofluoromethane	3.9	88-111	100		
acrolein	4.4	88-115	101		
1,1-dichloroethene	1.3	93-100	96	61-145	14
carbon disulfide	2.0	104-118	111		
iodomethane	2.1	91-104	98		
1,1,1-trichloro-2,2,2-trifluoroethane	3.6	90-112	101		
1,1,2-trichloro-1,2,2-trifluoroethane	2.1	99-113	106		
acetone	30	8-151	79		
3-chloropropene	3.3	99-121	110		
methylene chloride	3.3	87-107	97		
trans-1,2-dichloroethene	1.9	92-103	98		
acrylonitrile	2.8	94-111	103		
1,1-dichloroethane	1.1	103-110	106		
vinyl acetate	2.1	97-110	103		
cis-1,2-dichloroethene	2.1	97-110	103		
2-butanone	1.6	104-115	109		
chloroform	2.1	97-110	104		
1,1,1-trichloroethane	2.2	91-104	98		
carbon tetrachloride	1.9	93-104	99		
benzene	1.3	94-102	98	76-127	11
1,2-dichloroethane	1.8	98-109	104		
crotonaldehyde	3.1	85-103	94		
trichloroethene	6.5	77-114	96	71-120	14
dibromomethane	1.7	94-104	99		
1,2-dichloropropane	3.5	85-104	95		
bromodichloromethane	2.1	93-106	100		
2-chloroethyl vinyl ether	2.1	89-101	95		
cis-1,3-dichloropropene	2.1	97-111	104		
4-methyl-2-pentanone	0.29	97-99	98		
toluene	1.4	95-103	99	76-125	13
trans-1,3-dichloropropene	2.1	95-109	102		
1,1,2-trichloroethane	3.3	83-101	92		
ethylmethacrylate	1.4	96-104	100		
tetrachloroethene	0.83	89-94	91		
2-hexanone	4.1	86-110	98		
dibromochloromethane	2.6	87-102	94		
1,2-dibromoethane	3.4	88-108	98		

Table 5-10 (CONTINUED). Precision and Accuracy for Method 8240 Aqueous Volatiles Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
chlorobenzene	0.69	94-98	96	75-130	13
1,1,1,2-tetrachloroethane	3.6	90-111	101		
ethylbenzene	0.93	96-101	98		
m,p-xylenes	1.3	98-106	102		
o-xylene	1.6	98-107	103		
styrene	0.98	101-107	104		
bromoform	3.1	83-101	92		
cis-1,4-dichloro-2-butene	3.3	77-94	85		
1,2,3-trichloropropane	1.3	90-97	93		
1,1,2,2-tetrachloroethane	0.74	89-93	91		
trans-1,4-dichloro-2-butene	2.2	104-119	111		
1,2-dibromo-3-chloropropane	2.5	76-102	89		
Surrogates:					
D4-1,2-dichloroethane	0.79	111-114	113	76-114	NA
bromofluorobenzene	0.52	102-103	102	86-115	NA
D8-toluene	0.33	102-103	102	88-110	NA

Table 5-11. Precision and Accuracy for Method 624 Aqueous Volatiles Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
chloromethane	7.2	145-207	176	D-273	NA
vinyl chloride	2.8	135-155	145	D-251	NA
bromomethane	4.3	117-143	130	D-242	NA
chloroethane	6.8	92-124	108	14-230	NA
acrolein	3.4	17-163	90	*	NA
1,1-dichloroethene	4.0	89-108	99	D-234	NA
methylene chloride	6.1	83-112	98	D-221	NA
trans-1,2-dichloroethene	2.2	91-101	96	54-155	NA
acrylonitrile	4.4	D-188	91	*	NA
1,1-dichloroethane	1.9	91-100	96	59-155	NA
cis-1,2-dichloroethene	6.1	88-118	103	*	NA
chloroform	2.5	91-102	97	51-138	NA
1,1,1-trichloroethane	2.8	83-95	89	52-162	NA
carbon tetrachloride	3.2	81-95	88	70-140	NA
benzene	4.1	82-100	91	37-151	NA
1,2-dichloroethane	2.5	89-101	95	49-155	NA
trichloroethene	3.2	82-96	89	71-157	NA
1,2-dichloropropane	4.1	81-99	90	D-210	NA
bromodichloromethane	3.1	83-96	90	35-155	NA
2-chloroethyl vinyl ether	3.9	82-99	90	D-305	NA
cis-1,3-dichloropropene	4.2	80-98	89	D-227	NA
toluene	5.0	84-107	96	47-162	NA
trans-1,3-dichloropropene	3.2	80-93	87	17-183	NA
1,1,2-trichloroethane	2.2	87-97	92	52-150	NA
tetrachloroethene	5.4	82-106	94	64-148	NA
dibromochloromethane	3.2	83-97	90	53-149	NA
chlorobenzene	4.4	87-107	97	37-160	NA
ethylbenzene	4.7	85-106	95	37-162	NA
m,p-xylenes	5.9	85-113	99	*	N/A
o-xylene	5.8	87-115	101	*	NA
styrene	7.1	83-117	100	*	NA
bromoform	3.4	82-97	89	45-169	NA
1,1,2,2-tetrachloroethane	5.6	83-108	96	46-157	NA
Surrogates:					
D4-1,2-dichloroethane	NA**	NA	NA	76-114	N/A
bromofluorobenzene	NA	NA	NA	86-115	N/A
D8-toluene	NA	NA	NA	88-110	N/A

*Not target compounds for Method 624.
** Not available (NA)

Table 5-12. Precision and Accuracy for Method 625 Aqueous Volatiles Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
N-nitrosodimethylamine	6.2	39-47	43	*	N/A
bis(2-chloroethyl) ether	2.7	75-82	78	12-158	N/A
1,3-dichlorobenzene	3.2	76-84	80	D-172	N/A
1,4-dichlorobenzene	2.2	79-84	81	20-124	N/A
1,2-dichlorobenzene	2.4	78-84	81	32-129	N/A
bis(2-chloroisopropyl) ether	23.0	39-81	60	36-166	N/A
N-nitroso-Di-N-propylamine	7.4	76-95	86	D-230	N/A
hexachloroethane	3.2	71-78	75	40-113	N/A
nitrobenzene	1.2	81-84	83	35-180	N/A
isophorone	5.0	86-99	92	21-196	N/A
bis(2-chloroethoxy) methane	3.4	78-87	83	33-184	N/A
1,2,4-trichlorobenzene	5.5	79-93	86	44-142	N/A
naphthalene	2.7	80-87	84	21-133	N/A
hexachlorobutadiene	5.5	77-91	84	24-116	N/A
hexachlorocyclopentadiene	14.4	79-122	101	*	N/A
2-chloronaphthalene	16.6	22-37	30	60-118	N/A
dimethylphthalate	4.5	81-93	87	D-112	N/A
acenaphthylene	4.2	82-94	88	33-145	N/A
2,6-dinitrotoluene	4.4	82-94	88	50-156	N/A
acenaphthene	6.3	82-98	90	47-145	N/A
2,4-dinitrotoluene	7.0	53-66	59	39-139	N/A
diethylphthalate	6.8	84-103	94	D-114	N/A
4-chlorophenyl phenyl ether	6.4	82-100	91	25-158	N/A
fluorene	6.9	83-102	93	59-121	N/A
diphenylamine (N-nitroso)	4.9	81-94	88	*	N/A
1,2-diphenylhydrazine	6.1	85-102	93	*	N/A
4-bromophenyl phenyl ether	7.4	82-103	92	53-127	N/A
hexachlorobenzene	3.9	84-95	90	D-152	N/A
phenanthrene	5.0	87-101	94	54-120	N/A
anthracene	2.4	82-88	85	27-133	N/A
di-N-butylphthalate	2.5	85-92	89	1-118	N/A
fluoranthene	6.1	83-100	91	26-137	N/A
pyrene	11.5	76-107	91	52-115	N/A
benzidine	10.2	66-90	78	**	N/A
butylbenzylphthalate	6.1	93-112	102	D-152	N/A
3,3'-dichlorobenzidine	3.0	90-98	94	D-262	N/A
benzo(A)anthracene	11.3	22-171	146	33-143	N/A
bis(2-ethylhexyl)phthalate	5.9	84-100	92	8-158	N/A
chrysene	17.4	55-95	75	17-168	N/A
di-N-octylphthalate	5.6	85-100	93	4-146	N/A
benzo(B)fluoranthene	6.2	74-90	82	24-159	N/A
benzo(K)fluoranthene	4.9	84-98	91	11-162	N/A

*Not target compounds for Method 625.
**No limits were established in the method.

Table 5-12 (CONTINUED). Precision and Accuracy for Method 625 Aqueous Semivolatiles Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
benzo(A)pyrene	11.6	68-97	82	17-163	N/A
indeno(1,2,3-c,d)pyrene	8.2	70-89	79	D-171	N/A
dibenzo(a,h)anthracene	10.2	68-93	80	D-227	N/A
benzo(g,h,i)perylene	3.3	78-86	82	D-219	N/A
phenol	3.0	58-70	64	5-112	N/A
2-chlorophenol	4.1	90-115	102	23-134	N/A
2-nitrophenol	3.2	95-115	105	29-182	N/A
2,4-dimethylphenol	3.4	91-112	101	32-119	N/A
2,4-dichlorophenol	3.1	92-111	102	39-135	N/A
p-chloro-m-cresol	3.2	97-117	107	22-147	N/A
2,4,6-trichlorophenol	3.5	96-119	107	37-144	N/A
2,4-dinitrophenol	8.8	73-125	99	D-191	N/A
4-nitrophenol	7.4	38-59	48	D-132	N/A
4,6-dinitro-o-cresol	3.9	102-129	116	D-181	N/A
pentachlorophenol	4.2	90-117	104	14-176	N/A
Surrogates:					
D5-nitrobenzene (SS 1)	5.9	80-95	88	35-114	N/A
2-fluorobiphenyl (SS 2)	7.3	80-100	90	43-116	N/A
D14-terphenyl (SS 3)	6.7	82-100	91	33-141	N/A
D10-pyrene (SS 4)	6.7	49-60	54	40-130	N/A
2-fluorophenol (SS5)	4.0	69-88	79	21-100	N/A
D5-phenol (SS6)	3.1	58-69	63	10-94	N/A
2,4,6-tribromophenol (SS7)	4.5	90-118	104	10-123	N/A

Table 5-13. Precision and Accuracy for Various Inorganic and Wet Chemistry Methods

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
Water-CAWW 245.1, EPA CLP, 7470 SW-846			**Soil-CAWW 245.5, EPA CLP, 7471 SW-846**		
Mercury in Water and Soil					
Method: Chemical Analysis of Water and Wastes 245.1					
Water	1.8	101-112	106	75-125	20
Soil	3.8	91-119	105	75-125	20
Total Hardness as $CaCO_3$ in Water/Lachat 10-301-31-1-A					
Method: Chemical Analysis of Water and Wastes 130.1					
Water	0.88	100-106	103	75-125	20
Alkalinity					
Method: Chemical Analysis of Water and Wastes 310.2/Lachat 10-303-31-1-A					
Water	2.0	93-105	99	75-125	20
Chloride					
Method: Chemical Analysis of Water and Wastes 325.2/Lachat 10-117-07-1-A					
Water	0.32	100-102	101	75-125	20
Residue, Nonfilterable (Total Suspended Solids)					
Method: Chemical Analysis of Water and Wastes 160.2					
Water	11	76-146	111	NA	20
Residue, Filterable (Total Dissolved Solids)					
Method: Chemical Analysis of Water and Wastes 160.1					
Water	3.2	90-110	101	NA	20
Ammonia					
Method: Chemical Analysis of Water and Wastes 350.1/Lachat 10-107-06-1-A					
Water	0.62	95-99	97	75-125	20
Hexavalent Chromium					
Method: Lachat 10-124-13-1-A					
Water	2.1	97-111	104	75-125	20
Fluoride					
Method: Chemical Analysis of Water and Wastes 340.2/Lachat 10-109-12-2-A					
Water	0.96	103-109	106	75-125	20
Sulfate					
Method: Chemical Analysis of Water and Wastes 375.4/SW-846 9038					
Water	3.3	89-109	99	75-125	20

Table 5-13 (CONTINUED). Precision and Accuracy for Various Inorganic and Wet Chemistry Methods

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
Nitrate and Nitrite					
Method: Chemical Analysis of Water and Wastes 353.2					
Nitrate	0.22	97-98	98	75-125	20
Nitrite	0.29	114-116	115	75-125	20

Table 5-14. Precision and Accuracy for Radiochemistry Methods

Parameter		%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
					%R Limits	%RPD
Strontium-90						
Water		5.2	94-129	111	75-125	25
Soil		14	107-111	109	75-125	25
Gross Alpha and Beta						
Water	Alpha	5.2	51-94	73	75-125	25
Water	Beta	3.2	58-85	71	75-125	25
Soil	Alpha	0.99	39-52	46	75-125	25
Soil	Beta	1.6	56-71	64	75-125	25
Isotopic Uranium						
U238		5.8	67-107	87	75-125	25
U234		6.3	64-108	86	75-125	25
Radium-226						
Water		0.88	88-94	91	75-125	25
Total Radium						
Water		2.5	28-61	44	75-125	25

Table 5-15. Precision and Accuracy for Organic Characterization Methods

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
Total Organic Halides					
Soil	3.8	79-104	91	75-125	
Aqueous					
Total Organic Carbon					
Soil	0.71	99-103	101	75-125	
Aqueous					
Total Petroleum Hydrocarbons					
Aqueous					
Soil					
Oil and Grease					
Aquoeus	11	56-128	92	75-125	
Soil					

Table 5-16. Precision and Accuracy for Method 8010 Aqueous and Low Level Volatile Solid Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
Solid:					
chloromethane	26	67-189	128	D-193	20
vinyl chloride	10	86-147	112	28-163	20
bromomethane	2.1	93-105	99	D-144	20
chloroethane	3.9	85-109	97	46-137	20
1,1-dichloroethene	7.8	69-119	94	28-167	20
methylene chloride	5.8	127-152	140	25-162	20
trans-1,2-dichloroethene	4.3	90-115	102	38-155	20
1,1-dichloroethane	5.0	90-119	104	47-132	20
chloroform	4.3	94-119	106	49-133	20
bromochloromethane	6.4	81-119	100	49-133	20
1,1,1-trichloroethane	6.8	80-120	100	41-138	20
carbon tetrachloride	5.0	90-119	104	43-143	20
1,2-dichloroethane	5.0	90-119	104	51-147	20
trichloroethene	4.4	94-119	106	35-146	20
1,2-dichloropropane	7.4	88-129	108	44-156	20
bromodichloromethane	4.4	94-119	108	42-172	20
dibromomethane	4.4	94-119	108	42-172	20
2-chloroethyl vinyl ether	5.4	98-127	112	14-186	20
cis-1,3-dichloropropene	8.8	86-134	110	22-178	20
trans-1,3-dichloropropene	7.9	96-137	117	22-178	20
1,1,2-trichloroethane	5.4	98-127	113	39-136	20
tetrachloroethene	9.1	91-139	115	26-162	20
dibromochloromethane	5.4	98-127	112	24-191	20
1,2-dibromoethane	4.6	98-123	110	24-191	20
chlorobenzene	12	83-146	115	38-150	20
1,1,1,2-tetrachloroethane	4.4	94-119	106	38-150	20
bromoform	4.8	102-127	115	13-159	20
1,1,2,2-tetrachloroethane	6.8	80-120	100	8-184	20
1,2,3-trichloropropane	8.5	82-130	106	8-184	20
bromobenzene	4.4	94-119	106	8-184	20
2-chlorotoluene	4.4	94-119	106	8-184	20
4-chlorotoluene	5.4	98-127	112	8-184	20
1,3-dichlorobenzene	3.9	81-106	94	7-187	20
1,4-dichlorobenzene	3.9	81-106	94	42-143	20
1,2-dichlorobenzene	6.3	71-112	92	D-208	20
Surrogates:					
bromofluorobenzene	NA	NA	NA	69-123	N/A
trichlorofluoromethane	NA	NA	NA	76-135	N/A

Table 5-16 (CONTINUED). Precision and Accuracy for Method 8010 Aqueous and Low Level Solid Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
Aqueous:					
chloromethane	23	63-179	121	D-193	20
vinyl chloride	21	54-166	110	28-163	20
chloroethane	7.6	79-124	101	46-137	20
bromomethane	10	88-142	115	D-144	20
1,1-dichloroethene	7.3	83-125	104	28-167	20
methylene chloride	5.1	64-102	83	25-162	20
trans-1,2-dichloroethene	8.2	74-124	99	38-155	20
1,1-dichloroethane	14	65-145	105	47-132	20
chloroform	13	76-147	111	49-133	20
bromochloromethane	16	70-153	112	49-133	20
1,1,1-trichloroethane	17	67-160	114	41-138	20
carbon tetrachloride	12	79-144	112	43-143	20
1,2-dichloroethane	6.1	88-122	105	51-147	20
trichloroethene	6.7	98-132	115	35-146	20
1,2-dichloropropane	6.1	88-122	105	44-156	20
bromodichloromethane	6.7	98-132	115	42-172	20
dibromomethane	8.7	78-128	103	42-172	20
2-chloroethyl vinyl ether	8.2	74-124	99	14-186	20
cis-1,3-dichloropropene	6.3	82-120	101	22-178	20
trans-1,3-dichloropropene	5.4	92-122	108	22-178	20
1,1,2-trichloroethane	7.8	81-126	104	39-136	20
tetrachloroethene	6.1	88-122	105	26-162	20
dibromochloromethane	6.7	85-124	104	24-191	20
1,2-dibromoethane	7.4	83-125	104	24-191	20
chlorobenzene	6.7	98-132	115	38-150	20
1,1,1,2-tetrachloroethane	6.8	80-121	100	38-150	20
bromoform	14	85-155	120	13-159	20
1,1,2,2-tetrachloroethane	4.1	68-98	83	8-184	20
1,2,3-trichloropropane	5.5	62-102	82	8-184	20
bromobenzene	6.0	76-114	75	8-184	20
2-chlorotoluene	4.8	81-111	96	8-184	20
4-chlorotoluene	3.5	70-96	83	8-184	20
1,3-dichlorobenzene	2.4	85-101	93	7-187	20
1,4-dichlorobenzene	2.7	78-97	88	42-143	20
1,2-dichlorobenzene	1.5	82-93	88	D-208	20
Surrogates:					
bromofluorobenzene	NA	NA	NA	69-123	N/A
trichlorofluoromethane	NA	NA	NA	76-135	N/A

Table 5-17. Precision and Accuracy for Method 8020 Aqueous and Low Level Solid Samples, and Total Recoverable Petroleum Hydrocarbons as Gasoline by Modified Method 8015

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria %R Limits	%RPD
Solid:					
benzene	7.6	90-143	117	39-150	20
toluene	19	59-162	110	46-148	20
chlorobenzene	5.3	115-140	127	55-135	20
ethylbenzene	5.3	115-140	127	32-160	20
m-xylene	5.8	106-135	121	55-135	20
p-xylene	5.8	106-135	121	55-135	20
o-xylene	5.0	106-131	119	55-135	20
styrene	8.5	105-145	125	55-135	20
1,3-dichlorobenzene	4.9	106-131	119	50-141	20
1,4-dichlorobenzene	4.9	106-131	119	42-143	20
1,2-dichlorobenzene	9.5	95-143	119	37-154	20
Aqueous:					
benzene	1.4	92-101	96	39-150	20
toluene	1.7	84-95	90	46-148	20
chlorobenzene	2.8	87-104	96	55-135	20
ethylbenzene	3.0	86-105	95	32-160	20
m-xylene	1.6	85-96	90	55-135	20
p-xylene	1.6	85-96	90	55-135	20
o-xylene	2.3	84-99	92	55-135	20
styrene	1.6	88-99	94	55-135	20
1,3-dichlorobenzene	1.2	90-98	94	50-141	20
1,4-dichlorobenzene	1.9	87-100	94	42-143	20
1,2-dichlorobenzene	1.2	90-97	94	37-154	20
Total Recoverable Petroleum Hydrocarbons as Gasoline					
Soil (80° purge)	0.86	94-100	97	50-150	25
Soil (40° purge)	1.1	86-93	90	50-150	25
Medium Soil	2.8	102-117	109	50-150	25
Water	2.3	94-108	101	50-150	25

Table 5-18. Precision and Accuracy for Method 8270 Aqueous Semivolatile Organic Extractables Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
N-nitrosodimethylamine	8.5	76-122	99		
pyridine	2.0	87-97	92		
ethylmethacrylate	8.1	71-110	90		
paraldehyde	9.1	71-117	94		
2-picoline	2.7	92-123	107		
nitrosomethylethylamine	26	9-52	30		
methyl methane sulfonate	9.1	72-120	96		
N-nitrosodiethylamine	8.7	78-125	101		
ethyl methane sulfonate	8.2	80-126	103		
phenol	6.3	88-124	106	12-89	42
aniline	15	58-138	98		
pentachloroethane	7.5	64-97	81		
bis(2-chloroethyl) ether	7.9	82-126	104		
2-chlorophenol	7.8	81-123	102	27-123	40
1,3-dichlorobenzene	8.8	70-113	91		
benzyl chloride	8.8	66-107	86		
1,4-dichlorobenzene	7.8	68-105	87	36-97	28
benzyl alcohol	8.3	94-148	121		
1,2-dichlorobenzene	7.8	69-105	87		
2-methylphenol	8.0	82-127	105		
bis(2-chloroisopropyl) ether*	8.4	86-136	111		
3-methylphenol	4.7	68-114	91		
4-methylphenol	4.7	68-114	91		
N-nitrosopyrrolidine	6.8	85-123	104		
N-nitrosomorpholine	6.4	66-94	80		
acetophenone	7.1	87-129	108		
N-nitroso-di-n-propylamine	5.8	80-117	98	41-116	38
o-toluidine hydrochloride	7.4	52-122	87		
hexachloroethane	8.9	62-102	82		
nitrobenzene	8.6	70-113	92		
N-nitrosopiperidine	5.8	76-104	90		
isophorone	5.4	79-107	93		
2,4-dimethylphenol	7.8	69-106	87		
2-nitrophenol	5.2	72-96	84		
1,3,5-trichlorobenzene	5.0	24-42	33		
benzoic acid	7.8	211-324	268		
bis(2-chloroethoxy) methane	9.7	26-45	36		
o,o,o-triethylphosphorothioate	3.0	8-76	42		
2,4-dichlorophenol	8.0	59-92	76		
1,2,4-trichlorobenzene	5.9	60-82	71	39-98	28

*2,2'-oxybis (1-chloropropane)

Table 5-18 (CONTINUED). Precision and Accuracy for Method 8270 Aqueous Semivolatile Organic Extractables Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
naphthalene	5.7	70-95	82		
4-chloroaniline	2.5	85-98	91		
2,6-dichlorophenol	4.4	58-93	76		
o-phenylenediamine	3.0	46-55	51		
hexachlorobutadiene	5.7	49-67	58		
1,2,3-trichlorobenzene	5.5	65-88	76		
N-nitroso-di-n-butylamine	5.8	75-103	89		
p-chloro-m-cresol	5.5	88-118	103	23-97	42
p-phenylenediamine	7.0	15-22	18		
safrole	9.3	66-110	88		
m-phenylenediamine	18	31-87	59		
2-methylnaphthalene	5.2	91-120	106		
1-methylnaphthalene	6.3	129-182	155		
1,2,4,5-tetrachlorobenzene	6.1	48-94	71		
1,2,3,5-tetrachlorobenzene	6.1	48-94	71		
2,4,6-trichlorophenol	8.2	49-129	88		
2,4,5-trichlorophenol	3.4	70-103	87		
isosafrole	5.6	53-98	75		
2-chloronaphthalene	9.8	65-112	90		
1-chloronaphthalene	14	51-109	80		
1,2,3,4-tetrachlorobenzene	5.4	58-78	68		
2-nitroaniline	7.0	30-67	49		
1,4-napthoquinone	10	33-120	77		
1,4-dinitrobenzene	7.0	82-115	98		
dimethyl phthalate	6.6	75-108	92		
2,6-dinitrotoluene	6.6	73-105	89		
1,3-dinitrobenzene	6.6	80-114	97		
acenaphthylene	6.7	71-102	86		
3-nitroaniline	6.0	78-153	115		
acenaphthene	6.9	73-106	90	46-118	31
2,4-dinitrophenol	8.4	79-209	144		
4-nitrophenol	8.9	60-172	116	10-80	50
2,4-dinitrotoluene	8.0	74-114	94	24-96	38
dibenzofuran	6.0	76-105	90		
pentachlorobenzene	6.4	63-89	76		
2-naphthylamine	6.8	23-50	36		
1-naphthylamine	4.8	75-127	101		
2,3,4,6-tetrachlorophenol	4.9	69-118	94		
diethyl phthalate	6.6	78-112	95		
zinophos	6.7	77-111	94		
4-chlorophenyl phenyl ether	4.4	57-72	65		

Table 5-18 (CONTINUED). Precision and Accuracy for Method 8270 Aqueous Semivolatile Organic Extractables Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
fluorene	4.3	73-92	83		
4-nitroaniline	13	28-173	101		
5-nitro-o-toluidine	16	17-192	105		
1,2-diphenylhydrazine	6.4	98-139	119		
4,6-dinitro-2-methylphenol	9.5	64-199	131		
N-nitrosodiphenylamine*	7.8	55-134	94		
diphenylamine	7.3	57-132	94		
1,3,5-trinitrobenzene	11	43-163	103		
phenacetin	3.5	82-99	91		
4-bromophenyl phenyl ether	8.0	64-99	81		
diallate (trans isomer)	9.7	31-53	42		
dimethoate	14	41-92	67		
hexachlorobenzene	9.0	54-88	71		
4-aminobiphenyl	8.9	73-117	95		
pronamide	8.8	74-120	97		
pentachlorophenol	8.4	45-119	82	9-103	50
pentachloronitrobenzene	8.6	63-102	83		
phenanthrene	8.1	75-117	96		
anthracene	8.7	72-115	94		
di-n-butyl phthalate	6.9	80-116	98		
quinoline, 4-nitro, 1-oxide	10	D-230	104		
methapyrilene	7.9	49-122	85		
isodrin	8.7	73-118	95		
cyclophosphamide	10	39-70	55		
fluoranthene	11	62-113	88		
benzidine	11	45-85	65		
pyrene	6.1	81-113	97	26-127	31
aramite	11	52-98	75		
p-dimethylaminoazobenzene	5.7	81-111	96		
chlorobenzilate	7.9	82-127	105		
3,3'-dimethylbenzidine	18	5-198	101		
butylbenzyl phthalate	7.33	98-146	122		
2-acetylamino fluorene	6.0	98-136	117		
4,4'-methylene-bis(2-chloroaniline)	5.0	84-110	97		
3,3'-dichlorobenzidine	5.4	80-107	93		
dimethoxybenzidine	37	1-109	55		
bis(2-ethylhexyl) phthalate	23	51-219	135		
benzo(a)anthracene	7.4	77-115	96		
chrysene	5.1	78-103	90		

*Cannot be separated from diphenylamine.

Table 5-18 (CONTINUED). Precision and Accuracy for Method 8270 Aqueous Semivolatile Organic Extractables Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
di-n-octyl phthalate	7.6	85-129	107		
famphos	7.7	52-79	66		
benzo(b)fluoranthene	3.4	85-102	93		
7,12-dimethylbenzanthracene	5.1	58-76	67		
benzo(k)fluoranthene	10	62-107	84		
benzo(a)pyrene	5.3	71-95	83		
3-methylchloranthrene	10	29-52	41		
dibenzo(a,j)acridine	3.0	77-91	84		
indeno(1,2,3-c,d)pyrene	4.2	76-96	86		
dibenzo(a,h)anthracene	4.2	74-94	84		
benzo(g,h,i)perylene	5.4	72-96	84		
diallate (cis isomer)	7.9	32-50	41		
Surrogates:					
nitrobenzene-D5	NA	NA	NA	35-114	N/A
2-fluorobiphenyl	NA	NA	NA	43-116	N/A
terphenyl-D14	NA	NA	NA	33-141	N/A
phenol-D5	NA	NA	NA	10-94	N/A
2-fluorophenol	NA	NA	NA	21-100	N/A
2,4,6-tribromophenol	NA	NA	NA	10-123	N/A

NOTE: The following compounds did not recover well in the precision and accuracy studies and are, therefore, not listed in the tables: benzal chloride; alpha, alpha-dimethylphenethylamine; hexachloropropane; and benzotrichloride.

Table 5-19. Precision and Accuracy for Method 8270 Low Level Solid Semivolatile Organic Extractables Samples

Parameter	%RSD	3 σ Limits	Avg. % R	Method Acceptance Criteria	
				%R Limits	%RPD
N-nitrosodimethylamine	4.5	118-155	137		
pyridine	4.7	75-99	87		
ethylmethacrylate	6.3	79-116	98		
paraldehyde	2.2	92-105	98		
2-picoline	3.1	97-128	107		
nitrosomethylethylamine	3.2	27-33	30		
methyl methane sulfonate	4.4	62-81	71		
N-nitrosodiethylamine	3.4	104-128	116		
ethyl methane sulfonate	2.4	102-118	110		
phenol	3.8	118-148	133	26-90	35
aniline	7.4	47-74	60		
pentachloroethane	1.5	67-74	70		
bis(2-chloroethyl) ether	2.5	107-125	116		
2-chlorophenol	2.1	91-103	97	25-102	50
1,3-dichlorobenzene	0.46	80-82	81		
benzyl chloride	2.2	89-102	95		
1,4-dichlorobenzene	1.8	75-84	79	28-104	27
benzyl alcohol	2.8	108-128	118		
1,2-dichlorobenzene	0.72	79-83	81		
2-methylphenol	3.2	98-118	108		
bis(2-chloroisopropyl) ether*	3.7	214-267	241		
3-methylphenol	5.9	80-114	97		
4-methylphenol	5.9	80-114	97		
N-nitrosopyrrolidine	4.4	96-125	111		
N-nitrosomorpholine	4.1	101-129	4.1		
acetophenone	4.4	85-104	94		
N-nitroso-di-n-propylamine	4.1	103-132	117	41-126	38
o-toluidine hydrochloride	17	23-70	47		
hexachloroethane	1.7	84-93	89		
nitrobenzene	3.1	115-139	127		
N-nitrosopiperidine	2.4	100-116	108		
isophorone	2.4	120-139	130		
2,4-dimethylphenol	2.3	91-105	98		
2-nitrophenol	1.6	87-96	91		
1,3,5-trichlorobenzene	2.1	33-37	35		
benzal chloride	0.62	86-89	88		
benzoic acid	2.2	184-210	197		
bis(2-chloroethoxy) methane	6.7	79-119	99		
o,o,o-triethylphosphorothioate	1.7	68-76	72		
2,4-dichlorophenol	2.2	76-87	81		
1,2,4-trichlorobenzene	1.6	73-77	75	75	23

* 2,2'-oxybis (1-chloropropane)

Table 5-19 (CONTINUED). Precision and Accuracy for Method 8270 Low Level Solid Semivolatile Organic Extractables Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
naphthalene	1.5	89-97	93		
4-chloroaniline	6.0	35-50	42		
2,6-dichlorophenol	2.4	76-87	81		
o-phenylenediamine	2.8	55-65	60		
alpha,alpha-dimethyl phenethylamine	17	8-24	16		
hexachloropropene	4.4	37-48	43		
hexachlorobutadiene	4.6	56-73	64		
1,2,3-trichlorobenzene	0.88	72-76	74		
benzotrichloride	1.9	52-58	55		
N-nitroso-di-n-butylamine	3.2	112-136	124		
p-chloro-m-creosol	2.8	95-112	103	26-103	33
p-phenylenediamine	3.2	33-40	37		
safrole	0.97	78-83	81		
2-methylnaphthalene	1.2	113-122	117		
1-methylnaphthalene	1.3	165-178	172		
1,2,4,5-tetrachlorobenzene	2.8	70-83	77		
1,2,3,5-tetrachlorobenzene	2.8	70-83	77		
2,4,6-trichlorophenol	0.51	91-94	93		
2,4,5-trichlorophenol	1.2	83-89	86		
isosafrole	0.52	96-99	98		
2-chloronaphthalene	2.3	95-109	102		
1-chloronaphthalene	3.6	70-87	78		
1,2,3,4-tetrachlorobenzene	1.6	74-81	78		
2-nitroaniline	4.0	16-20	18		
1,4-napthoquinone	2.2	11-12	11		
1,4-dinitrobenzene	2.8	97-115	106		
dimethyl phthalate	0.50	93-95	94		
2,6-dinitrotoluene	1.6	95-105	100		
1,3-dinitrobenzene	2.7	103-122	112		
acenaphthylene	0.30	97-99	98		
3-nitroaniline	3.6	87-108	97		
acenaphthene	1.4	91-99	95	31-137	19
2,4-dinitrophenol	3.1	102-122	112		
4-nitrophenol	3.7	98-123	110	11-114	50
2,4-dinitrotoluene	1.9	123-138	131	28-89	47
dibenzofuran	0.50	87-89	88		
pentachlorobenzene	2.0	72-81	77		
2-naphthylamine	7.1	14-22	18		
1-naphthylamine	4.8	29-39	34		
2,3,4,6-tetrachlorophenol	4.8	72-96	84		
diethyl phthalate	1.4	93-101	97		
zinophos	2.6	114-133	124		

Table 5-19 (CONTINUED). Precision and Accuracy for Method 8270 Low Level Solid Semivolatile Organic Extractables Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
4-chlorophenyl phenyl ether	0.29	72-73	73		
fluorene	1.0	86-91	89		
4-nitroaniline	3.5	83-102	92		
5-nitro-o-toluidine	7.4	37-96	66		
1,2-diphenylhydrazine	24	21-132	76		
4,6-dinitro-2-methylphenol	2.7	109-128	119		
N-nitrosodiphenylamine*	2.4	95-110	102		
diphenylamine	2.4	95-110	102		
1,3,5-trinitrobenzene	3.1	109-132	121		
phenacetin	4.5	101-133	117		
4-bromophenyl phenyl ether	2.4	81-94	88		
diallate (trans isomer)	4.0	40-51	46		
dimethoate	5.2	80-110	95		
hexachlorobenzene	2.7	74-87	80		
4-aminobiphenyl	6.0	44-64	54		
pronamide	3.0	84-101	93		
pentachlorophenol	1.8	78-87	83	17-109	47
pentachloronitrobenzene	2.6	91-106	98		
phenanthrene	2.1	93-105	99		
anthracene	2.0	88-98	93		
di-n-butyl phthalate	3.0	102-121	111		
quinoline, 4-nitro, 1-oxide	6.8	51-78	67		
methapyrilene	7.2	111-116	113		
isodrin	1.8	87-98	93		
cyclophosphamide	3.1	48-58	53		
fluoranthene	2.9	84-100	92		
pyrene	8.7	44-75	59	35-142	36
p-dimethylaminoazobenzene	7.0	49-75	62		
chlorobenzilate	8.5	55-92	73		
3,3'-dimethylbenzidine	3.4	21-26	23		
butylbenzyl phthalate	8.0	54-88	71		
2-acetylamino fluorene	2.3	61-70	66		
4,4'-methylene-bis(2-chloroaniline)	3.4	39-47	43		
3,3'-dichlorobenzidine	2.6	42-49	45		
bis(2-ethylhexyl) phthalate	8.2	51-84	67		
benzo(a)anthracene	3.4	54-66	60		
chrysene	5.8	46-65	56		
di-n-octyl phthalate	9.4	98-175	137		
famphos	36	D-85	41		
benzo(b)fluoranthene	2.5	88-102	95		
7,12-dimethylbenzanthracene	4.4	86-112	99		
benzo(k)fluoranthene	6.2	71-103	87		

*Cannot be separated from diphenylamine.

Table 5-19 (CONTINUED). Precision and Accuracy for Method 8270 Low Level Solid Semivolatile Organic Extractables Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
benzo(a)pyrene	2.0	85-95	90		
3-methylchloranthrene	2.1	87-98	93		
dibenzo(a,j)acridine	2.2	90-103	96		
indeno(1,2,3-c,d)pyrene	1.2	75-81	78		
dibenzo(a,h)anthracene	1.5	90-99	94		
benzo(g,h,i)perylene	2.4	75-87	81		
diallate (cis isomer)	3.7	40-50	45		
Surrogates:					
nitrobenzene-D5	NA	NA	NA	23-120	N/A
2-fluorobiphenyl	NA	NA	NA	30-115	N\A
terphenyl-D14	NA	NA	NA	18-137	N/A
phenol-D5	NA	NA	NA	24-113	N/A
2-fluorophenol	NA	NA	NA	25-121	N/A
2,4,6-tribromophenol	NA	NA	NA	19-122	N/A

NOTE: The following compounds did not recover well in the precision and accuracy study and are, therefore, not listed in the tables: benzidine, aramite, and dimethoxybenzidine.

Table 5-20. Precision and Accuracy for Method 8270 Medium Level Solid Semivolatile Organic Extractables Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
N-nitrosodimethylamine	12	43-92	67		
pyridine	14	38-97	68		
ethylmethacrylate	14	42-100	71		
paraldehyde	14	43-105	74		
2-picoline	14	39-93	66		
nitrosomethylethylamine	14	11-26	18		
methyl methane sulfonate	12	34-72	53		
N-nitrosodiethylamine	15	39-99	69		
ethyl methane sulfonate	13	43-95	69		
phenol	8.8	49-83	66	26-90	35
aniline	11	43-84	63		
pentachloroethane	12	48-104	76		
bis(2-chloroethyl) ether	12	46-99	73		
2-chlorophenol	14	41-101	71	25-102	50
1,3-dichlorobenzene	15	44-113	78		
benzyl chloride	13	42-92	67		
1,4-dichlorobenzene	14	45-110	78	28-104	27
benzyl alcohol	15	34-91	63		
1,2-dichlorobenzene	14	45-110	78		
2-methylphenol	13	40-92	66		
bis(2-chloroisopropyl) ether	11	48-96	72		
3-methylphenol	12	43-91	67		
4-methylphenol	12	43-91	67		
N-nitrosopyrrolidine	14	39-99	69		
N-nitrosomorpholine	15	39-100	69		
acetophenone	12	45-98	72		
N-nitroso-di-n-propyl amine	12	46-95	71	41-126	38
o-toluidine hydrochloride	9.6	46-84	65		
hexachloroethane	13	44-102	73		
nitrobenzene	11	49-100	75		
N-nitrosopiperidine	14	44-108	76		
isophorone	14	43-91	67		
2,4-dimethylphenol	13	45-99	72		
2-nitrophenol	16	42-117	80		
1,3,5-trichlorobenzene	14	24-59	41		
benzal chloride	13	45-105	75		
benzoic acid	17	183-543	363		
bis(2-chloroethoxy) methane	10.0	168-311	239		
o,o,o-triethylphosphorothioate	12	42-91	66		
2,4-dichlorophenol	18	31-102	66		

Table 5-20 (CONTINUED). Precision and Accuracy for Method 8270 Medium Level Solid Semivolatile Organic Extractables Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
1,2,4-trichlorobenzene	15	46-127	86	38-107	23
naphthalene	14	46-110	78		
4-chloroaniline	13	46-102	74		
2,6-dichlorophenol	12	45-99	72		
o-phenylenediamine	1.3	108-117	113		
alpha,alpha-dimethyl phenethylamine	46	D-45	19		
hexachloropropene	13	43-92	70		
hexachlorobutadiene	14	48-121	85		
1,2,3-trichlorobenzene	15	46-125	86		
benzotrichloride	14	42-103	73		
N-nitroso-di-n-butylamine	13	48-105	76		
p-chloro-m-cresol	14	45-113	79	26-103	33
p-phenylenediamine	16	48-135	91		
safrole	16	46-130	88		
m-phenylediamine	11	16-31	24		
2-methylnaphthalene	14	60-152	106		
1-methylnaphthalene	14	84-213	149		
1,2,4,5-tetrachlorobenzene	14	45-111	78		
1,2,3,5-tetrachlorobenzene	14	45-111	78		
hexachlorocyclopentadiene	15	13-35	24		
2,4,6-trichlorophenol	15	42-108	75		
2,4,5-trichlorophenol	13	45-105	75		
isosafrole	15	42-111	77		
2-chloronaphthalene	14	40-96	68		
1-chloronaphthalene	20	34-139	87		
1,2,3,4-tetrachlorobenzene	14	44-109	76		
2-nitroaniline	12	23-50	37		
1,4-napthoquinone	13	30-70	50		
1,4-dinitrobenzene	19	32-114	73		
dimethyl phthalate	16	42-108	75		
2,6-dinitrotoluene	14	41-103	72		
1,3-dinitrobenzene	17	36-113	74		
acenaphthylene	16	37-106	72		
3-nitroaniline	13	45-106	75		
acenaphthene	16	38-108	73	31-137	19
2,4-dinitrophenol	15	49-134	91		
4-nitrophenol	12	26-54	40	11-114	50
2,4-dinitrotoluene	15	38-103	71	28-89	47
dibenzofuran	16	38-105	71		
pentachlorobenzene	16	42-116	79		
2-naphthylamine	13	19-42	30		
1-naphthylamine	10	44-85	65		
2,3,4,6-tetrachlorophenol	17	40-121	81		

Table 5-20 (CONTINUED). Precision and Accuracy for Method 8270 Medium Level Solid Semivolatile Organic Extractables Samples

Parameter	%RSD	3 σ Limits	Avg. %R	Method Acceptance Criteria	
				%R Limits	%RPD
diethyl phthalate	17	37-110	73		
zinophos	16	37-103	70		
4-chlorophenyl phenyl ether	15	36-97	66		
fluorene	14	41-101	71		
4-nitroaniline	13	39-88	64		
5-nitro-o-toluidine	14	40-98	69		
1,2-diphenylhydrazine	15	39-100	69		
4,6-dinitro-2-methylphenol	14	56-133	94		
N-nitrosodiphenylamine*	10	54-98	76		
diphenylamine	10	54-98	76		
1,3,5-trinitrobenzene	11	56-107	81		
phenacetin	9.0	48-83	65		
4-bromophenyl phenyl ether	12	54-111	82		
diallate (trans isomer)	12	50-106	78		
dimethoate	11	41-80	60		
hexachlorobenzene	11	54-106	80		
4-aminobiphenyl	7.9	54-88	71		
pronamide	11	47-94	70		
pentachlorophenol	15	41-110	76	17-109	47
pentachloronitrobenzene	9.1	59-103	81		
phenanthrene	12	50-107	78		
anthracene	13	46-103	74		
di-n-butyl phthalate	13	47-103	75		
quinoline, 4-nitro, 1-oxide	9.2	80-141	110		
methapyrilene	13	41-96	69		
isodrin (van)	11	47-92	69		
cyclophosphamide	14	27-64	45		
fluoranthene	16	38-107	73		
benzidine	20	18-67	43		
pyrene	20	32-122	77	35-142	36
p-dimethylaminoazobenzene	17	38-114	76		
chlorobenzilate	14	43-106	75		
3,3'-dimethylbenzidine	20	18-69	43		
butylbenzyl phthalate	14	44-113	79		
2-acetylamino fluorene	13	41-92	67		
4,4'-methylene-bis(2-chloroaniline)	15	41-106	74		
3,3'-dichlorobenzidine	13	46-103	74		
dimethoxybenzidine	98	41-76	59		
bis(2-ethylhexyl) phthalate	17	38-122	80		

*Cannot be separated from diphenylamine.

Table 5-20(CONTINUED). Precision and Accuracy for Method 8270 Medium Level Solid Semivolatile Organic Extractables Samples

Parameter	%RSD	3 σ Limits	Avg. % R	Method Acceptance Criteria	
				%R Limits	%RPD
benzo(a)anthracene	11	51-104	78		
chrysene	18	36-119	77		
di-n-octyl phthalate	22	28-132	80		
famphos	17	17-52	34		
benzo(b)fluoranthene	22	20-103	62		
7,12-dimethylbenzanthracene	23	26-136	81		
benzo(k)fluoranthene	54	D-312	118		
benzo(a)pyrene	15	42-107	75		
3-methylchloranthrene	14	44-106	75		
dibenzo(a,j)acridine	9.3	56-99	77		
indeno(1,2,3-c,d)pyrene	9.0	57-99	78		
dibenzo(a,h)anthracene	10	54-104	79		
benzo(g,h,i)perylene	7.7	59-94	76		
diallate (cis isomer)	10	53-101	77		
Surrogates:					
nitrobenzene-D5	NA	NA	NA	23-120	
2-fluorobiphenyl	NA	NA	NA	30-115	
terphenyl-D14	NA	NA	NA	18-137	
phenol-D5	NA	NA	NA	24-113	
2-fluorophenol	NA	NA	NA	25-121	
2,4,6-tribromophenol	NA	NA	NA	19-122	

NOTE: The compound aramite did not recover well in the precision and accuracy study and is, therefore, not listed in the table.

6.0 Sample Receipt/Custody

CompuChem does not collect samples for its clients. However, we do provide glassware with collection procedures if the client needs this service. CompuChem meets program-specific shipping requirements and offers a patented SampleSaver shipping container. Glassware supplied by CompuChem for sample collection is purchased as cleaned and certified from the vendor. The glassware is cleaned according to the U.S. EPA Office of Solid Waste and Emergency Response (OSWER) directive published in the document *Specifications and Guidance for Contaminant-Free Glassware*. Analysis is performed by the vendor using low detection limit EPA methods. The certificates of analysis that accompany the glassware are reviewed and approved by the QA department before each lot of glassware is purchased. The certificates are filed in the QA department.

6.1 Quality Control of the VOA Storage Cabinet

Glassware that is purchased for sample collection is stored at CompuChem's warehouse facility. The bottles to be used for collecting samples for volatiles analysis are stored in a cabinet containing five to eight trays filled with activated carbon to prevent contamination. The charcoal is replaced monthly by the warehouse staff. A logbook is maintained to document when the charcoal is changed.

6.2 VOA Storage Stability Tests

Walk-in Cooler: Samples received for volatiles analysis are stored in the volatile walk-in cooler in the Sample Control department until the production planner requests these samples for preparation and analysis. To monitor volatile cross-contamination in the walk-in cooler, eight bottles filled with deionized water were initially placed on a tray in the cooler for four weeks. After this four-week incubation period, two bottles were removed from the cooler and tested by volatile GC/MS analysis using criteria from the most current U.S. EPA CLP SOW protocols to judge the acceptability of the results. Two freshly filled bottles were then placed at the back of the tray. Since this initial test, the front two bottles have been routinely removed for analysis and two new bottles placed in the back of the tray. In this way, the bottles are incubated for four weeks before testing. If the first of the two bottles for the week fails acceptance criteria, a holding blank known to be in the walk-in cooler is analyzed. If the holding blank also fails criteria, then the second bottle is scheduled for analysis. If the second bottle also fails acceptance criteria, then the walk-in cooler carbon filter is changed by the Facilities department staff.

Volatile GC/MS and GC Laboratory temporary storage units: To detect possible cross-contamination in the laboratory temporary storage units, testing is performed as described above for the walk-in cooler with the exception that four bottles from the GC Laboratory temporary storage unit are aged for two weeks and analyzed by GC

rather than GC/MS. The temporary storage units also have trays of activated carbon which are changed if testing indicates contamination.

6.3 Warehouse Pure Water System

Specific QC procedures are in place to ensure the integrity of the warehouse pure water system.

Quality Control of Warehouse Pure Water System

The warehouse has a Millipore pure water system which generates Type I reagent grade water. This laboratory pure water is used for trip blanks in SampleSavers, and, on occasion, for equipment/rinsate or field blank water provided as a service to various clients.

The pure water drawn from the Millipore pure water system is held in a series of five, 45-liter glass carboys. When a carboy is filled by the pure water system, QC tests must be run to document that the water is contaminant-free. A standard operating procedure describes the manner in which the water purity is verified for each of the carboys.

Upon installation of the Millipore/carboy system, a study was undertaken to verify that the carboys were contaminant-free and the pure water was stable for at least 30 days. While the system is only vulnerable to contamination by volatiles, certain semivolatiles (phthalates), and certain trace metals, the study included pesticide parameters as well. No target analytes were detected in any of the analyses. All carboys were approved for use and carboy 1 was characterized as "active" while the remaining carboys were labeled "back-up."

Procedure Description

When the first carboy is nearly drained, it is filled by the Millipore system and is in the test phase. The second carboy is then "active" while carboys 3-5 are "back-up" (and are used until the active carboy is drained). The test carboy is not to be used until notification from the QA department that the QC tests have met criteria.

When carboy 2 is nearly drained, it is filled and tested in the same manner. Ideally, all carboys will contain pure water, and only one carboy will be in the test phase at any given time. When water is not being used for a prolonged period of time, four carboys will be designated "back-up" while one will be "active." The carboy is tested at the time of filling for volatiles, semivolatiles, trace metals, and pesticides.

QC Water Purity Tests

All samples bottles used to check water purity are vendor-supplied cleaned and certified glassware. All glassware used is cleaned according to the OSWER directive, *Specifications and Guidance for Contaminant-Free Sample Glassware*.

QC samples are drawn for the following tests: volatile GC/MS, semivolatile GC/MS, trace metals analysis, and pesticides. Four QC check samples are drawn from the test carboy which include two 40-ml bottles for volatiles analysis, two one-liter glass bottles for semivolatiles analysis, two one-liter plastic bottles for trace metals analysis, and two one-liter glass bottles for pesticides analysis. Volatile QC check samples are placed in zip-lock bags for transportation to the laboratory.

The warehouse clerk records the label identification, method of analysis, and sample collector's initials in the water purity system logbook. When the information becomes available, the CompuChem identification (ID) number, the test status (approved or rejected), and the date results are received from the QA department are also recorded in the logbook.

The bottles are sent to CompuChem's Sample Control department, and assigned a CompuChem ID number, then sent directly to the appropriate sample preparation and/or testing laboratories. The turnaround time (TAT) in the LIMS is two days, indicating that the tests must be performed and reported directly to the QA department within 48 hours of sample receipt. The data is reviewed and approved by the analyst before being reported to QA.

The laboratory performs the volatiles analysis by U.S. EPA 10/92 SAM GC/MS methods. The instrument blank analyzed during the 12-hour calibration period in which the test sample is run must meet the following criteria:

- Methylene chloride must be < 2.0 μg/L.
- Acetone must be < 5.0 μg/L.
- Other target compound list (TCL) analytes must be < the contract required quantitation limit (CRQL).
- Tentatively identified compounds (TICs) must be < 2.0 μg/L.

A copy of the instrument blank must be sent to the QA department along with the QC test sample results. The following criteria are used for volatile test sample approval:

- Methylene chloride must be < 2.0 μg/L.
- Acetone must be < 5.0 μg/L.
- Other target compound list (TCL) compounds must be < the CRQL.
- TICs (those non-TCLs not present in the instrument blank and not known laboratory artifacts) must be < 10% of the nearest internal standard peak height.

The laboratory performs the semivolatiles analysis by methods set forth in U.S. EPA 10/92 SAM with the modification that a separatory funnel extraction is used to reduce the turnaround time. The method blank extracted with the QC

test sample must meet the following criteria:

- TCL phthalates must be < 5.0 µg/L.
- Other TCL analytes must be < the CRQL.
- TICs must be < 10.0 µg/L.

A copy of the method blank data must be sent to the QA department along with the QC test sample results. The following criteria are used for test sample approval:

- TCL phthalates must be < 5.0 µg/L.
- Other TCL analytes must be < the CRQL.
- TICs (those not present in the method blank and not known laboratory artifacts) must be < 10.0 µg/L.

The laboratory performs the pesticides analysis by U.S. EPA 10/92 SAM.The method blank extracted along with the QC test sample must meet the following criterion:

- All TCL analytes must be < the CRQL.

The following criterion is used for test sample approval:

- All TCL analytes must be < the CRQL.

The inorganic test sample is analyzed for all U.S. EPA CLP metals analyses. Note that inorganic test samples do not have to be digested before analysis, and the calibration blank analyzed before the test sample must not contain any of the target analytes above the contract-required detection limit (CRDL). All metals must be < the CRDL and not greater than two times the calibration blank level (for an analyte detected above the instrument detection limit [IDL] in the calibration blank).

As stated above, test results are sent directly to the QA department. It is the responsibility of the Production Planning and Control (PP&C) department coordinator to post the analytical queues once the data are approved by the analyst and turned in to the QA department. The QA department records the test outcome on a PC spreadsheet in the QA administrative office. When all tests have been completed on a certain carboy, the QA coordinator communicates the results by phone within 24 hours to the warehouse.

If the criteria for all analytical tests are met, the test carboy status is changed to *active*. If these criteria are exceeded, the supervisor of the Sample Control department is contacted and the unacceptable test rescheduled using the second bottle with the same sample ID. If contamination is confirmed by analysis of the second bottle, the QA specialist verifies that initial and retest data are comparable. If the data are comparable, the QA specialist rejects the test sample and requests the warehouse supervisor to empty the carboy. The corrective action taken must be documented in the warehouse water purity system logbook.

6.4 SampleSaver Preparation

A SampleSaver is a patented sample collection container provided as a service to customers who request that CompuChem supply bottles to the field. Orders are taken by a Customer Service department representative, who enters the orders into the LIMS. The LIMS then generates a worklist for each order. The SampleSaver worklist contains the following information:

- address of the client
- type of SampleSaver to be sent
- special instructions (e.g., use of COC form)
- method of shipment
- account number
- latest shipping date
- analysis codes for samples
- SampleSaver number assigned by the LIMS

The SampleSaver number is pre-printed on an adhesive label which is attached to the sample container by Sample Control personnel. An information packet is included, and consists of the client information sheet, instructions for using SampleSaver materials (these vary according to the type of SampleSaver that is sent), sample collection procedures (sent with all types of SampleSavers), a COC record, COC seals, sample ID labels, and return address labels. When soil samples are to be imported into the U.S., the U.S. Department of Agriculture (U.S.D.A.) requires that the sample containers be labeled with "Restricted Entry" labels. CompuChem encloses these labels and instructions for their use in SampleSavers sent outside the U.S. CompuChem has a U.S.D.A. permit to move imported soil samples.

SampleSaver configurations required by clients may include a preservative kit or laboratory pure water. Aqueous volatile trip blanks are preserved with HCl at the time of bottle preparation and before shipment from the laboratory. If the client requests that the COC record originates from CompuChem, a glassware release COC is used. The manager of subcontract administration and warehouse systems or designee signs and dates the record, which initiates the COC process. The SampleSaver is sealed with COC tape.

While the protocol for sample collection is left to the discretion of the field sampling crew, for samples requiring compliance to the Florida Department of Environmental Regulation sampling protocol, the use of blue ice for sampling preservation is discouraged and is adequate only if the samples have been precooled with wet ice. In addition, Florida and New Jersey both require that formal COC start when the precleaned sample containers are dispatched to the field from the laboratory. Refer to Section 7.0 for additional sample custody requirements.

The configuration of a SampleSaver is dependent on the client's needs. Each available configuration has a code that is listed on the SampleSaver worklist. To make up these configurations, the warehouse systems manager keeps on hand the necessary glassware for SampleSavers. Maintaining this glassware stock requires that the warehouse maintain an adequate supply of precleaned and precertified glassware.

Sample collection volume requirements and guidelines are listed in Table 6-1. If samples are received by the laboratory with insufficient volume to allow the laboratory to adhere to method volume requirements, a laboratory policy is in place to address their processing of samples. We will contact our client, who has the option to cancel analyses or to instruct CompuChem to proceed with analysis using a reduced volume. For example, when only one liter bottle of aqueous sample is received and the method requires 1000 ml for sample preparation, 500 ml are aliquoted for preparation and 500 ml are reserved as backup. This is done as a precaution in case of a laboratory accident during sample processing or the need to confirm a sample matrix effect through a repeat preparation and analysis. At the same time that the extraction volume is reduced for a low volume sample, the surrogate standard solution and final extract concentration volumes may be reduced proportionally. By doing so, elevation of detection limits is avoided. However, in meeting specific requirements of the U.S. EPA CLP, an adjustment in final extract volume would not occur, thereby resulting in elevated detection limits. In handling limited volume samples, our clients' instructions will be followed in achieving the desired CRQLs.

Table 6-1. Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes per Clean Water Act, 40 CFR 136 (1990), *Federal Register*

Parameter	Preservation	Holding Time[A] (days)	Containers polyethylene (P) glass (G)	Volume (ml)
acidity	cool, 4 °C	14	P or G	200
alkalinity	cool, 4 °C	14	P or G	100
ammonia	cool, 4 °C	28	P or G	500
biochemical oxygen demand	cool, 4 °C	2	P or G	1000
bromide	none required	28	P or G	200
chemical oxygen demand	cool, 4 °C add H_2SO_4 to pH < 2	28	P or G	100
chloride	none required	28	P or G	100
chlorine (total residual)	none required	0[B]	P or G	500
chromium VI	cool, 4 °C	1	P or G	500
coliform, fecal and total	cool, 4 °C	6 hours	P or G	200
	add 0.008% $Na_2S_2O_3$	2	P or G	500
color	cool, 4 °C			
cyanide (total)	cool, 4 °C add NaOH to pH > 12 add 0.6 g ascorbic acid[D]	14[C]	P or G	1000
cyanide amenable to chlorination (free)	cool, 4 °C add NaOH to pH > 12 add 0.6 g ascorbic acid[D]	14[C]	P or G	500
fluoride	none required	28	P	500
gross alpha/beta	HNO_3 to pH < 2	180	P or G	2000/100
gamma	HNO_3 to pH < 2 cool, 4 °C			2000
total radium	HNO_3 to pH < 2 cool, 4 °C	180	P or G	4000
radiological soils	4 °C	180	P or G	8 oz

[A] from time of sample collection
[B] Zero days implies that the sample must be analyzed immediately.
[C] reduced to 24 hours if sulfide is present unless sulfide is removed before preservation
[D] used only in the presence of residual chlorine

Table 6-1 (CONTINUED). Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes per Clean Water Act, 40 CFR 136 (1990), *Federal Register*

Parameter	Preservation	Holding Time^ (days)	Containers polyethylene (P) glass (G)	Volume (ml)
hardness	Add HNO_3 to pH < 2. Add H_2SO_4 to pH < 2.	180	P or G	250
hydrogen ion (pH)	none required	0	P or G	40
Kjeldahl nitrogen	cool, 4 °C Add H_2SO_4 to pH < 2.	28	P or G	1000
mercury	Add HNO_3 to pH < 2.	28	P or G	500
metals (except Cr VI, Hg)	Add HNO_3 to pH < 2.	180	P or G	500
nitrate	cool, 4 °C	2	P or G	100
nitrate-nitrite	cool, 4 °C Add H_2SO_4 to pH < 2.	28	P or G	500
oil and grease	cool, 4 °C	28	G	1000
organic carbon (total)	Add H_2SO_4 to pH < 2. cool, 4 °C	28	P or G	100
organic nitrogen (total)	Add HCL or H_2SO_4 to pH < 2. cool, 4 °C	28	P or G	500
phenols (total)	Add H_2SO_4 to pH < 2. cool, 4 °C	28	G	1000
phosphorus (elemental)	Add H_2SO_4 to pH < 2. cool, 4 °C	2	G	500
phosphorus (total)	cool, 4 °C Add H_2SO_4 to pH < 2.	28	P or G	200
solids (total)	cool, 4 °C	7	P or G	100
solids (filterable)	cool, 4 °C	7	P or G	100
solids (non-filterable)	cool, 4 °C	7	P or G	200
solids (settleable)	cool, 4 °C	2	P or G	1000
silica	cool, 4 °C	28	P or G	100
specific conductance	cool, 4 °C	28	P or G	250
sulfate	cool, 4 °C	28	P or G	250
sulfide	cool, 4 °C Add zinc acetate and NaOH to pH > 9	7	P or G	

^ from time of sample collection

Table 6-1 (CONTINUED). Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes per Clean Water Act, 40 CFR 136 (1990), *Federal Register*

Parameter	Preservation	Holding Time[A] (days)	Containers polyethylene (P) glass (G)	Volume (ml)
sulfite	none required	0	P or G	250
surfactants	cool, 4 °C	2	P or G	250
turbidity	cool, 4 °C	2	P or G	250
purgeable halocarbons	cool, 4 °C Add 0.008% $Na_2S_2O_3$[D]	14	G	80
purgeable aromatic hydrocarbons	cool, 4 °C Add 0.008% $Na_2S_2O_3$,[D] HCl to pH ≤ 2.[C]	14	G	80
phenols	cool, 4 °C Add 0.008% $Na_2S_2O_3$.[D]	7/40[E]	P or G	250
benzidines	cool, 4 °C Add 0.008% $Na_2S_2O_3$.[D]	7/7[E,F]	P or G	2000
phthalate esters	cool, 4 °C Add 0.008% $Na_2S_2O_3$.[D]	7/40[E]	P or G	2000
nitrosamines	cool, 4 °C, dark Add 0.008% $Na_2S_2O_3$.[D]	7/40[E]	P or G	2000
polychlorinated biphenyls	cool, 4 °C	7/40[E]	P or G	2000
nitroaromatics and isophorone	cool, 4 °C, dark Add 0.008% $Na_2S_2O_3$[D]	7/40[E]	P or G	2000
polynuclear aromatic hydrocarbons	cool, 4 °C Add 0.008% $Na_2S_2O_3$.[D]	7/40[E]	P or G	2000
haloethers	cool, 4 °C Add 0.008% $Na_2S_2O_3$	7/40[E]	P or G	2000
chlorinated hydrocarbons	cool, 4 °C	7/40[E]	P or G	2000
TCDD	cool, 4 °C Add 0.008% $Na_2S_2O_3$.[D]	7/40[E]	P or G	2000
pesticides	cool, 4 °C, pH 5-9	7/40[E]	P or G	2000

[A] from time of sample collection
[C] If acrolein/acrylonitrile are to be analyzed, preserve to pH of 4-5.
[D] only used in the presence of residual chlorine
[E] to complete extraction/to complete analysis following extraction
[F] if stored under inert, oxidant-free conditions

Table 6-2. Requirements for Containers, Preservation[A], Holding Times, and Recommended Sample Volumes per U.S. EPA CLP SOW for Inorganics Analysis 3/90 and U.S. EPA CLP SOW for Organics Analysis 3/90 and EPA 10/92 SAM

Parameter	Preservation	Holding Time[D] (days)	Containers polyethylene (P) glass (G)	Volume (ml)
INORGANICS:				
cyanide (total and amenable to chlorination)	cool, 4 °C Add NaOH to pH > 12, and 0.6 g ascorbic acid.[H]	12	P or G	1000
metals (except Hg)	Add HNO_3 to pH < 2	180	P or G	500[B]
mercury	Add HNO_3 to pH < 2	26	P or G	500[B]
ORGANICS:				
aqueous volatiles	4 °C (± 2 °C)[F] Add HCl to pH ≤ 2.	10 [G]	G-TLSSL[C]	80
soil/sediment volatiles	4 °C (± 2 °C)[F]	10	G-TLC[C]	4 oz
aqueous semivolatiles	4 °C (± 2 °C)[F]	5/40 [E]	G[C]	2000
soil/sediment, semivolatiles	4 °C (± 2 °C)[F]	10/40[E]	G[C]	8 oz
aqueous pesticides/PCBs	4 °C (± 2 °C)[F]	5/40[E]	G[C]	2000
soil/sediment pest./PCBs	4 °C (± 2 °C)[F]	5/40[E]	G[C]	8 oz

[A] Water samples only; preservation performed by sampler immediately upon sample collection. Soil/sediment samples are maintained at 4 °C until analysis. Dissolved metals samples are filtered on site by sampler before addition of preservative.

[B] can be combined into a one-liter bottle

[C] All containers are one-liter glass bottles or 8-oz jars with Teflon-lined cap except aqueous volatiles (G-TLSSL: 40-ml glass bottle with Teflon-lined, septum-sealed lid), and soil/sediment volatiles (4-oz glass jar with Teflon-lined cap, G-TLC).

[D] from validated time of sample receipt (VTSR)

[E] to complete extraction/to complete analysis following extraction

[F] Preserve samples at time of collection. Samples should be stored in the dark until extraction/analysis.

[G] until analysis

[H] used only in the presence of residual chlorine

Table 6-3. Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes for Organics per *Test Methods for Evaluating Solid Waste Physical/Chemical Methods SW-846* (U.S. EPA, third edition)

Parameter	Preservation	Holding Time[A] (days)	Containers[B] polyethylene (P) glass (G)	Volume
VOLATILE ORGANICS:				
liquids (8010, 8020, 8240)	cool, 4 °C Add four drops of concentrated HCl.[C]	14	G (40-ml)	80 ml
soil/sediments/sludge (8010, 8020, 8240)	cool, 4 °C	14	G (4-oz)	4 oz
concentrated wastes	none	14	G (4-oz or 8-oz)	4 oz
EXTRACTABLE ORGANICS:				
liquids (8080, 8140, 8150, 8270, 8280)	cool, 4 °C[D]	7/40	G (1-liter) amber	2000 ml
extractable soil/sediments/sludges	cool, 4 °C	14/40	G (8-oz)	8 oz
concentrated wastes	none	14/40	G (8-oz)	8 oz
METALS: (Except Cr VI and Hg)				
total recoverable	Add HNO_3 to pH < 2.	180	P or G	500/200 ml/g
dissolved	Filter on site and add HNO_3 to pH < 2.	180	P or G	500/200 ml/g
suspended (TBD)	Filter on site.	180	P or G	500/200 ml/g
total	Add HNO_3 to pH < 2.	180	P or G	500/200 ml/g
CHROMIUM VI	cool, 4 °C	1	P or G	500/200 ml/g
MERCURY	Add HNO_3 to pH < 2.	28	P or G	500/200 ml/g
DISSOLVED	Filter and add HNO_3 to pH < 2.	28	P or G	500/200 ml/g

[A] from time of sample collection

[B] For volatile liquid samples, glass (G) 40-ml bottles with Teflon-lined septum-sealed lids are used. For volatile solids or wastes, glass (G) 4-oz jar with Teflon-lined caps are used. For extractable liquid and solid samples, glass (G) one-liter bottles or 8-oz jars with Teflon-lined caps are used.

[C] If using the preservative kit provided by CompuChem, use 10-12 drops of 30% HCl solution. If residual chlorine is present, collect sample in a 4-oz soil VOA container that has been prepreserved with four drops of 10% sodium thiosulfate. Intermediate vessel may not be used. Add sample to vial with $Na_2S_2O_3$. Fill one half to one third full, add acid, complete filling one vial. If acrolein/acrylonitrile are to be analyzed, preserve to pH of 4-5.

[D] If residual chlorine present, add 1 ml of 10% sodium thiosulfate per liter.

Table 6-4. Minimal Volume Requirements for Full U.S. EPA CLP or Appendix IX

Analysis	Soils	Soil QC	Waters	Waters QC[A,B]
Volatiles	4-oz x 1	[C]	40-ml x 3	40-ml x 2
Semivolatiles, pesticides/ PCBs, metals, mercury, cyanide, phenols, dry weight	4-oz x 2 OR 8-oz x 1	4-oz x 2 OR 8-oz x 1	1 L SV x 2 1 L P/PCB x 2 1 L Met/Hg x 1[D] 1 L CN x 1[D]	1 L SV x 2 1 L P/PCB x 2 1 L Met/Hg x 1[D] 1 L CN x 1[D]
Wet chemistry (Appendix IX sulfides)	NA	NA	1 L x 2	1 L x 3

[A] This is additional volume needed for sample designated *Use QC*. Add this to volumes specified in column on the left.

[B] This does not apply to U.S. EPA 10/92 SAM (low concentration) since no laboratory spikes or duplicates required.

[C] Sufficient volume already in original sample's four-ounce jar; additional volume not needed.

[D] Will need an additional liter if both *dissolved* and *total* are required.

6.5 Preparation and Shipping of Preservative Kits

Preservative kits are prepared and sent with SampleSavers whenever the type of analysis requested by a customer requires the addition of specified preservatives to the collected sample. The SampleSaver code that appears on the LIMS worksheet determines the configuration of the preservatives to be shipped in the kit. A preservative kit contains:

- four-ounce wide mouth glass jars (Allpak #2043 with a #5212 green phenolic Teflon-lined cap) of 30% nitric acid, 30% sulfuric acid, 30% hydrochloric acid, 10N sodium hydroxide solution, and/or 2N zinc acetate (Baker Instra-analyze Trace Metal Grade Reagent)
- 25-30 jumbo transfer pipets (Fisher Scientific #13-678-8)
- pH paper canister (Hydrion jumbo vials, wide range #4800)
- instructions for the proper use of preservatives

Each jar of preservative is prepared in the Inorganics Sample Preparation Laboratory and labeled with the reagent lot number, preparation date, and expiration date (one year). The jars are sent to the off-site warehouse where lot numbers are recorded in a logbook. There the jars are sealed with Teflon tape and placed into plastic bags. The jars are then placed into tin cans and packed with vermiculite, sealed, labeled, and stored in the warehouse chemical cabinet. The chemicals are pulled as needed for distribution into preservative kits.

7.0 Sample and Hardcopy Data Custody and Control

For a sample or for hardcopy data generated from analyzing a sample to be handled according to legal COC requirements, it must be:

- in the physical possession of an authorized field or laboratory staff member, or authorized transferee, or
- after physical possession of an authorized staff member, in the staff member's view, or
- secured (after physical possession) to prevent tampering, or
- placed in a designated secure area with restricted access.

Any change of possession or custody is documented on a COC form (Figure 7-1), and must include the names of individuals relinquishing and receiving the sample or data. Because individuals use their initials, the Technical Communications department maintains a list of signatures that includes the initials, full names, and employee identification numbers of all individuals signing COCs. Full signatures are required for all work performed for the New Jersey Department of Environmental Protection and Energy (NJDEPE). The date and time of transfer is also noted. Any corrections to COC information are made by drawing a single horizontal line through the incorrect entry, and printing the correct entry adjacent to the original entry. All corrections are initialled and dated.

Depending on client and regulatory requirements, sample COC may begin when pre-cleaned sample containers are sent from the laboratory to the field, when the sample containers are filled in the field and loaded into the shipping coolers, or when samples arrive at the laboratory. The NJDEPE and the Florida Department of Environmental Regulation require that we initiate COC at the laboratory when loading pre-cleaned empty sample collection containers into SampleSavers before shipping them to the field (see also Section 6). The warehouse shipping and receiving clerk is responsible for initiating the COC in these cases.

When sample COC is initiated in the field by a CompuChem client, the person responsible for initiating COC in the laboratory is the receiving clerk. The receiving clerk signs and dates the COC form. The samples are then assigned unique, sequential six-digit identification numbers by the LIMS (as described in Section 7.2.2).

Once the receiving clerk has logged in and documented the receipt of the sample, the sample is relinquished to the sample custodian on duty. The sample custodians and the supervisor of the Sample Control department have keys that unlock the sample storage coolers. Samples are filed in walk-in coolers until laboratory staff request specific samples by completing internal COC forms or batch sheets (Figure 7-2). The internal COCs are completed the same way, and the sample custodian relinquishes the samples to the laboratory staff member. The internal COC form is used to document the sample's movement from the custodian to the analyst to final disposition.

The sample custodian is responsible for purging raw samples from cold storage at the prescribed time. Unused raw samples are stored in a controlled temperature environment for 60 days after data submission to the client. NJDEPE samples are stored for one year. Sample report dates are documented in the LIMS. Sample labels are color coded and placed in the cooler by date of receipt, allowing bottles to be easily retrievable from the storage unit shelves. once segregated by the sample custodian, the hazardous waste technician completes the preparation for discarding

the identified samples for hazardous waste disposal.

Each laboratory area has its own planner from the Production Planning and Control (PP&C) department. Daily worklists are generated from the LIMS to assist PP&C staff in scheduling samples for preparation or analysis. The planner prioritizes and batches samples according to their holding times or due date. The person who prepares or analyzes the sample accepts possession of the sample. Samples are transferred by cart, under COC, from the walk-in cooler to the laboratory area in which the samples are needed. (Samples that must be preserved by refrigeration do not remain unrefrigerated for more than two hours.)Some methods require that samples be processed at room temperature; this may require that samples be unrefrigerated for more than two hours. When the samples have been analyzed, the extracts (for extractable portions of the sample) are kept in a locked freezer (if required) under custody of the sample custodian on duty.

The LIMS schedules the appropriate analyses for samples and automatically tracks the progress of samples through the laboratory. The custody of a sample may be determined at any time by reviewing the scheduling details within the LIMS. Signatures and employee ID numbers on the internal COCs, sample preparation and analytical worksheets, and sequence runlogs are used as a paper trail to document the physical transfer of the samples, and to document exactly who handled the samples at each stage of processing.

The integrity of the samples in the laboratory is assured by the building security, which is controlled by an electronic card entry system. The exterior doors and the doors of restricted access interior areas are equipped with card readers. Each CompuChem employee has an entry card with a photo ID that must be visibly displayed on their clothing. An employee's entry card is coded to allow access only to those areas that he/she needs to enter to do his/her job. The card entry system also generates a record of the movements, or attempted movements, of every employee throughout the building.

Hardcopy reports are stored and numbered to maintain strict document control. The document control clerk maintains an inventory of all hardcopy data stored. Hardcopy data are filed according to case and sample delivery group (SDG) number. The data are stored both at an off-site warehouse and in the laboratory in a secured area accessible by authorized entry only. The document control clerk is responsible for properly storing data in both locations. If hardcopy data must be removed from storage for audits or data inquiries, CompuChem uses formal document control procedures to ensure that the hardcopy data is correctly removed from and returned to storage.

7.1 Electronic Data Custody and Control

The mainframe and minicomputer systems at CompuChem are secured by using assigned log-on accounts and individual passwords. The Systems and Laboratory Automation department assigns and approves log-on accounts once the user has completed a computer access form that has been signed by the user's department manager. Passwords expire and must be changed every 30 days; they cannot be reused for one year after they expire.

Menu options are available to authorized users only, and are controlled by software that uses local attributes. These local attributes are created and maintained by the computer operations analyst. Users are allowed access only to those portions of the systems that are necessary for them to do their jobs. Each user is restricted to certain menus and options through his/her log-on

account, and only authorized staff have editing capabilities.

The entire LIMS is *shadowed* on a second, identical mainframe computer, and the power to the computer system is linked to an uninterrupted power supply. All data sent through the mainframe and minicomputers are backed up incrementally each day. Additional weekly, monthly, and yearly backup and archiving procedures are performed in compliance with the Operations Systems SOPs. Tapes are stored in a restricted, secured area within the facility. Access to the storage area is limited to Systems and Laboratory Automation department staff, Facilities department staff, and laboratory managers. The storage area is air-conditioned and kept free of debris. Tapes are retained for at least five years, unless clients specifically request a longer or shorter retention period.

Controlling software and hardware means testing and validating it before using it in production. These tests are planned and executed following SOPs. These SOPs describe documentation procedures that we use and records that we maintain on newly developed and revised software. A test set of data, with manually calculated or previously validated results, is used to verify performance of the new or revised software routine or hardware configuration. The Systems and Laboratory Automation department manager must approve the evaluated test results before releasing the product. Backup copies of old software versions are retained for 90 days before being removed from the system. The Systems and Laboratory Automation department uses a software package that automatically controls and structures software maintenance, development, and production. The program logs all actions into an audit trail database.

Numerous forms, worksheets, and sequence runlogs are generated from the computer systems and include analytical worksheets and the sample record. Individual laboratory non-analytical SOPs contain examples of these forms with instructions for completing them. Analytical results are reported on certain form templates either through direct electronic transfer from the instrument, indirect transfer via a local area network (LAN) linked to the instrument, or through manual data entry. All three mechanisms have specific security and QC features that are described in detail in the PP&C and Systems and Laboratory Automation department SOPs. Some data are reported electronically to clients using computer diskettes.

The case auditor in the Report Preparation department follows a checklist to verify that each case is complete. The checklist requires that the case auditor verify that a diskette has been produced (when required), that a copy has been made for long-term archival, and that the hardcopy and diskette data are identical. For certain clients, a second diskette is produced, which is in a format compatible with Contract Compliance Screening (CCS) software, an automated diskette data validation program used by these clients. CompuChem also uses a CCS software routine for both organics and inorganics U.S. EPA CLP diskette data validation before releasing either the hardcopy or electronic data.

7.2 Logging in Samples

Bench Procedures

The following steps are completed for all samples as they are received by CompuChem. If a sample requires special handling upon receipt, the manager of PP&C is consulted for instructions on properly handling and documenting the sample. A letter of receipt (LOR) and completed COC forms are sent to all clients 24-48 hours after the sample is received

at CompuChem.

Before opening and while inspecting each sample, employees wear protective clothing (laboratory coats, safety glasses, and gloves). These items are worn at all times when working in defined areas. Once the radiological survey is completed, containers are received into the facility. Each sample container is inspected before it is opened to make sure that it has not been damaged or opened during shipment. Any padlocks, sealing tape, or custody seals on the samples are inspected to make sure that they are intact, and any observations are recorded on the COC form. If the custody seals, tapes, or padlocks are broken, the commercial client is contacted through Customer Service or the Sample Management Office for EPA samples for permission to continue processing.

Each container is opened under the fume hood in the Sample Control department and checked for breakage. Vials containing samples to be analyzed for volatile compounds are checked to ensure that there is no headspace or air bubbles. Sample identification information on the bottles is compared to the Traffic Reports (TRs), packing lists, and COC form included in the container. Any discrepancies are noted on the COC form by the receiving clerk. The Customer Service department notifies commercial clients (non-EPA) if there are discrepancies, and the supervisor of the Sample Control department notifies the SMO for discrepancies with EPA samples.

Sample Control department personnel accept custody of samples by signing and dating the COC form. Incoming EPA samples are checked against the Sample Management Office (SMO) receipt schedule. Depending on whether the client is the U.S. EPA or a commercial entity, samples are logged onto an EPA Receiving Log Sheet or a Commercial Receiving Log Sheet. The following items, where applicable, are noted on the sheet:

- case number
- CompuChem ID number (CC#)
- client name or order number
- field ID (sample ID)
- receiving date (RD)
- sampling date (SD)
- residual chlorine and sulfide check (cyanide only)
- matrix
- temperature
- analysis codes
- volume received
- pH (inorganics only)*
- SampleSaver number

*Aqueous volatile sample pH is taken after analysis and documented in the data report.

The condition of the refrigerant (whether any ice remains or whether the cooling packs are solid) is checked and the temperature of a representative sample (liquid samples only) is ascertained by wrapping a temperature strip around the outside of the container. The temperature is recorded on the Sample Record and on the Receiving Log Sheet. Residual chlorine and sulfide test strips are used for cyanide samples; results are indicated on the Receiving Log Sheet. When it is apparent through these checks that a sample was not properly preserved, the client is notified and a standard QA Notice is completed and placed in the sample file.

On each EPA COC, TR, and commercial client COC that is complete and correct, the statement "Received in Good Condition" is written or stamped, initialled, and dated by the receiving clerk. This statement indicates that the sample or group of samples were received intact with correct sample tags or custody seals (if applicable), pH (applicable to inorganic samples), and corresponding documentation. It does not, however, mean that the sample temperature fell within the recommended range 4°C ± 2°C; EPA samples are commonly received at temperatures above that range. The temperature is noted on the sample log sheet and on the sample record or gray envelope (for EPA samples).

Each log sheet and COC is reviewed by the Sample Control department supervisor who ensures that all information is properly documented. Each is stamped as having been reviewed, initialled, and dated.

Computer Log-in Procedures

EPA sample account information is entered by the EPA Project Manager or designee into the marketing portion of the LIMS to generate order and requisition numbers and to assign analysis codes. Analysis codes correspond to the procedure requested by the client, include the QC requirements, and define the analytical tasks that must be performed to satisfy method requirements. An EPA Scheduling Log is also completed to document order entry. Order and requisition numbers with analysis codes for all other samples are provided to receiving personnel by a Customer Service department account representative after the Receiving Log Sheet is reviewed and the sample information is entered into the LIMS for tracking.

As information for each sample is received into the LIMS, a CompuChem number (CCN) is generated. A CCN is a unique, six-digit laboratory identifier. It is added to the accessioning log sheet and to the COC (adjacent to the associated field ID number when possible). Sample labels containing the CCN are generated from the LIMS in numerical sequence. Each sample is labeled by wrapping the bottle with its unique computer-generated label, leaving as much of the field label exposed as possible. The sample labels are color-coded, and colors are rotated every two weeks by the supervisor of the Sample Control department or designee. Rotating the colored labels helps sample custodians to locate and purge sample bottles, extracts, or digestates after the required storage-to-disposal period has passed.

Once labeled, samples are transferred to the locked walk-in cooler. Samples to be analyzed for extractable components and inorganics are stored separately from samples to be analyzed for purgeable components. Standards are stored in separate refrigerated units in the analytical laboratories.

Worksheets used for sample analysis are generated from the LIMS for EPA organics analyses and for commercial clients. Worksheets for QC samples are also generated from the LIMS, but are printed on green paper. File folders are used to assemble field and QC sample information for report preparation. Green folders identify QC samples. Every folder contains a sample record generated when a sample is logged in and is used to document completion of each step of the analytical requirements. EPA sample file folders contain the Sample Record, and a gray envelope contains all information for the case including the yellow copy of the Organic Traffic Report (OTR), a copy of the COC, an original air bill, a copy of the sample log sheet, a copy of the EPA Scheduling Log,

custody tags if received, and a gray envelope contents sheet. The white copy of the OTR is returned with the cover sheet to the EPA SMO. Commercial sample file folders contain a Sample Record, a customer sample order information sheet, and a copy of the COC record.

Sample Transfer

Samples are occasionally received from the EPA that should have been sent to another laboratory. The procedure for transferring such samples consists of four steps.

1. A CompuChem COC form is filled out using the information from the sample tags.

2. Custody is relinquished to the courier by signing and dating the "Relinquished by" section of the COC form.

3. A copy of the COC form is maintained for the record. The original and all paperwork received with the samples are sent to the designated laboratory.

4. Notation is made on the TR that samples are being sent to another laboratory.

Receipt of Samples Hand-Delivered after Business Hours

When samples are hand-delivered after business hours, the actual date of sample receipt is recorded on the COC form. The sample's condition, the date, and the time of receipt are recorded on the organics and inorganics TRs. The notation "HD" (Hand Delivered) is made on the COC form on the TRs. The date of the following calendar day is recorded on the COC form when the sample is logged in. Samples are received into the LIMS using the date the sample log-in date.

Sample Processing after Receipt

Figure 7-3 depicts baseline configuration of data flow from sample receipt to release of the final report to the client.

COMPUCHEM
ENVIRONMENTAL
CORPORATION
3306 Chapel Hill/Nelson Highway
Research Triangle Park, NC 27709
1-800-833-5097

CHAIN-OF-CUSTODY RECORD

№ 0322

Ship to:	Project Name:	Field Point-of-Contact:
	Sampler Name:	Telephone No:
		Sampling for project complete? Y or N (See Note 1.)
Carrier: Airbill No.:	Sampler Signature:	Project-specific (PS) or Batch (B) QC: ____

Box #1: 1. Surface Water, 2. Ground Water, 3. Leachate, 4. Rinsate, 5. Soil/Sediment/Sludge, 6. Trip Blank, 7. Oil, 8. Waste, 9. Other: _____

Box #2: A. HCl, B. HNO_3, C. $NaHSO_4$, D. $Na_2S_2O_3$, E. Ice Only, O. Other: _____, N. Not Preserved

Box #3: F. Filtered, U. Unfiltered

Box #4: C. CLP 2/90, S. SW-846 -, W. CWA 600-series, L. Low Conc. CLP, R. Radiological, T. TCLP, O. Other

Box #5: H - High, M - Medium, L - Low

Sample ID (Organics: 9 characters max, Inorganics: 6 characters; see Note 2)	Date: Year: 19__	Time	Box #1 Matrix	Box #2 Preservative	Box #3 Filtered/Unfiltered	Box #4 Method	Box #5 Expect. Conc.	No. of Bottles	Use for Lab QC (MS or DUP)	Organics Analysis: VOA-GC/MS	SV-GC/MS	Pest/PCB-GC	Herb-GC	VOA-GC	Inorganics: Metals	Mercury	Cyanides	Other: Radiologicals	TOC/TOX	O&G/TPH	Phenols	Other	Remarks/Comments
	/	:																					
	/	:																					
	/	:																					
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Client's Special Instructions:

Lab: Received in Good Condition? Y or N Describe Problems, If Any:

#1 Relinquished By: (Sig.)	Date:	#2 Relinquished By: (Sig.)	Date:	#3 Relinquished By: (Sig.)	Date:	Sample storage time requested?____ (in days, see Note 3)
Company Name:	Time:	Company Name:	Time:	Company Name:	Time:	
#1 Received By: (Sig.)	Date:	#2 Received By: (Sig.)	Date:	#3 Received By: (Sig.)	Date:	DESTROY or RETURN data after five years of archival? (Circle choice, see Note 4)
Company Name:	Time:	Company Name:	Time:	Company Name:	Time:	

Note (1): If "N" lab will hold samples to await remainder of project-maximizing batch size and minimizing QC ratio, if "Y" lab will begin processing batches now. Note (2): If CLP inorganics diskette required, ID limited to maximum of six characters
Note (3): Samples stored 60 days after data report mailed at no extra charge. Note (4): All lab copies of data destroyed after five years unless client requests and pays for return of copies; annual storage fee billed in January of year six.

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Figure 7-2. Internal COC Form

COMPUCHEM ENVIRONMENTAL CORPORATION

Internal Chain-of-Custody

Laboratory: _____ **Requested By:** _____

Samples For: 1 2 3 (circle one) **Time Requested:** _____

Date Requested: _____

Check Where Applicable:

EPA ____ Water ____

Commercial ____ Soil ____

	CompuChem #'s	Bottle	Containers
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			
19.			
20.			

RELINQUISHED BY: _____ **RECEIVED BY:** _____

RELINQUISHED BY: _____ RECEIVED BY: _____

RELINQUISHED BY: _____ RECEIVED BY: _____

COMMENTS

_____

_____

Environmental Flow Diagram
May 26, 1992

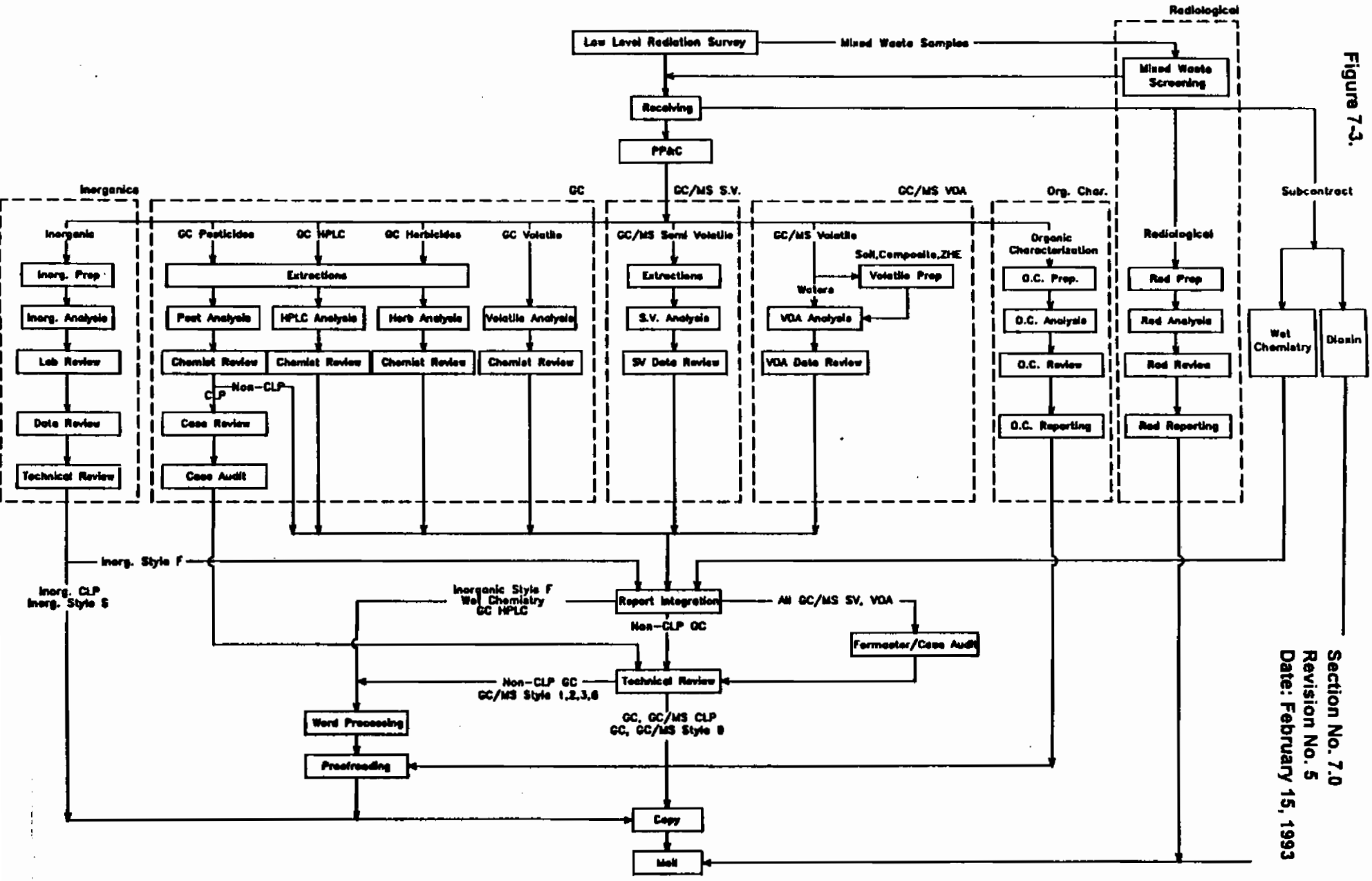

Figure 7-3.

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8.0 Calibration/Frequency

Instruments must be calibrated and recalibrated at regular intervals as specified by the applicable method, and consistent with the manufacturer's recommendations. The nature and frequency of such checks are specified in the analytical SOPs describing the instrumental analyses performed at CompuChem. Specific method calibration requirements are followed if more stringent than those listed here. The laboratory maintains records of all calibrations, recalibrations, and in-service checks of instruments. All calibrations are traceable to primary standards of measurement. Where the concept of traceability of measurement to primary standards is not applicable, the laboratory provides satisfactory evidence of correlation or accuracy of test results. Tables 8-1 through 8-13 list method-specific initial and continuing calibration requirements for all laboratories.

In the GC/MS Laboratory, the initial and continuing calibration data are stored on the instrument computer and are also accessible through the mainframe computer network. For methods published by the U.S. EPA CLP and in *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods SW-846*, the response factors from the calibrations are transferred automatically across the network, and downloaded onto the method-required forms. For non-CLP methods, hardcopies generated at the instrument are included in the data report if requested by the client.

All GC/MS calibration standard data include a summary of: (1) the variation (expressed as %RSD) of the response factors from each compound at each standard level in the initial calibration (multipoint), and (2) the variation (expressed as %D) in the multipoint compared with the response factor from the continuing calibration standard.

The hardware tuning and calibration of the instruments are documented in the instrument runlogs kept at the bench. If an instrument fails tuning or calibration criteria, hardware adjustments or other appropriate maintenance are performed and documented, and the analyst repeats the tuning and calibration attempt. Mass assignment is adjusted on GC/MS instruments using FC43 only as needed when tune or mass calibration criteria cannot be met successfully. The GC/MS volatile tuning compound, 4-bromofluorobenzene (BFB), or the semivolatile tuning compound, decafluorotriphenylphosphine (DFTPP), precedes the analytical sequence. These activities, if successful, are noted in the runlog and sample analyses may proceed. If not, corrective action may include additional maintenance by the full-time dedicated instrumentation staff, as described in Section 10.0.

Measurement devices, such as balances and thermometers, are calibrated against NIST- or EPA-certified sources. A certificate of the calibration is maintained with the laboratory manager and with QA records. All balances are calibrated daily or with each use using certified Class-S weights, and quarterly by the balance vendor as part of the service contract. All thermometers are traceable to either the Standards Laboratory's NIST-certified thermometer or to the certificate of NIST traceability supplied by the vendor. In-house calibration records are maintained by the senior standards chemist. This annual calibration verification is described in greater detail in the QA SOP manual. pH meters are calibrated daily or before being used with three certified buffer solutions ranging between 2.0 and 10.0 pH units. The range will bracket the test measurement target. The meter is set to a pH 7.0 buffer and calibrated with pH 2.0, 4.0, and 10.0. Acceptance criteria for the three certified buffer solutions is ±0.05 pH units.

Table 8-1. Initial Calibration Procedures for Analytical Equipment in the GC/MS Laboratory

Method	Tuning Frequency	Tuning Criteria	No. Stds. For Init. Calib.	Calibration Frequency & Concentration Range (CR)	Acceptance Criteria
624	once/12 hr	see Table 8.2	3	When continuing calibration (CC) fails CR: 40-160 µg/L	%RSD < 35% for all target compounds
625	once/12 hr	see Table 8.5	3	When CC fails CR: acid 50-150 µg/L BN: 25-100 µg/L	% RSD <35% for all target compounds
SW-846 8270	once/12 hr	see Table 8.5	5	When CC fails CR: 20-160 µg/L	%RSD < 30% (Appendix 3) for calibration check compounds (CCC); Minimum average response factor (RF) = 0.050 for system performance check compounds (SPCC)
SW-846 8240	once/12 hr	see Table 8.2	5	When CC fails CR: 20-200 µg/L co-eluting xylenes are added at half concentration	%RSD <30% for CCC (Appendix 4) minimum average RF = 0.300 for SPCC; 0.250 for bromoform
CLP 3/90 VOA	once/12 hr	see Table 8.3	5	When CC fails CR: 10-200 µg/L (with each co-eluting xylene and dichloro-ethene isomer at equal concentration)	%RSD ≤ 20.5 for most compounds; minimum RF specified (Appendix 5)

Table 8-1 (continued). Initial Calibration Procedures for Analytical Equipment in the GC/MS Laboratory

Method	Tuning Frequency	Tuning Criteria	No. Stds. For Init. Calib.	Calibration Frequency & Concentration Range (CR)	Acceptance Criteria
10/92 SAM low concentr. VOA	once/12 hr	see Table 8-3	5	When CC fails, CR: ketones 5-125 µg/L, all others 1-25 µg/L	%RSD ≤ 30 for most compounds; minimum RF specified (Appendix 6).
CLP 3/90 SV	once/12 hr	see Table 8-4	5	When CC fails, CR: 10-80 µg/L	%RSDs for all compounds and minimum RF criteria must be met. (Appendix 5)
10/92 SAM low concen-tration SV	once/12 hr	see Table 8-4	5	When CC fails, CR: 5-80 µg/L or 20-120 µg/L (compound specific)	%RSDs for all compounds and minimum RF criteria must be met. (Appendix 6)

Table 8-2. Bromofluorobenzene (BFB) Key Ions and Abundance Criteria for Methods 624 and 8240

Mass	Ion Abundance Criteria
50	15.0 - 40.0 % of the base peak
75	30.0 - 60.0 % of the base peak
95	base peak, 100% relative abundance
96	5.0 - 9.0 % of the base peak
173	< 2.0 % of mass 174
174	> 50.0 % of the base peak
175	5.0 - 9.0 % of mass 174
176	> 95.0 % but less than 101.0 % of mass 174
177	5.0 - 9.0 % of mass 176

Table 8-3. Bromofluorobenzene (BFB) Key Ions and Abundance Criteria for CLP 3/90 SOW and 10/92 SAM

Mass	Ion Abundance Criteria
50	8.0 - 40.0% of mass 95
75	30.0 - 66.0% of mass 95
95	base peak, 100% relative abundance
96	5.0 - 9.0% of mass 95 *
173	< 2.0% of mass 174
174	50.0 - 120.0 % of mass 95
175	4.0 - 9.0% of mass 174
176	93.0 - 101.0 percent of mass 174
177	5.0 - 9.0% of mass 176

* All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundances of m/z 174 may be up to 120% that of m/z 95.

Table 8-4. DFTPP Key Ions and Abundance Criteria for CLP 3/90 SOW and 10/92 SAM

Mass	Ion Abundance Criteria
51	30.0 - 80.0% of mass 198
68	< 2.0% of mass 69
69	present
70	< 2.0% of mass 69
127	25.0 - 75.0% of mass 198
197	< 1.0% of mass 198
198	base peak, 100% relative abundance*
199	5.0 - 9.0% of mass 198
275	10.0 - 30.0% of mass 198
365	> 0.75% of mass 198
441	present but < mass 443
442	40.0 - 110% of mass 198
443	15.0 - 24.0% of mass 442

* All ion abundances must be normalized to m/z 198, the nominal base peak, even though the ion abundances of m/z 442 may be up to 110% that of m/z 198.

Table 8-5. DFTPP Key Ions and Abundance Criteria for Methods 625 and 8270

Mass	Ion Abundance Criteria
51	30.0 - 60.0% of mass 198
68	< 2.0% of mass 69
69	Present
70	< 2.0% of mass 69
127	40.0 - 60.0% of mass 198
197	< 1.0% of mass 198
198	base peak, 100% relative abundance
199	5.0 - 9.0% of mass 198
275	10.0 - 30.0% of mass 198
365	> 1.00% of mass 198
441	present but < mass 443
442	> 40.0% of mass 198
443	17.0 - 23.0% of mass 442

Table 8-6. Continuing Calibration Procedures for Analytical Equipment in the GC/MS Lab.

Method	Frequency of Cont. Calib. & Concentration Level (CL)	Acceptance Criteria	Sources for Standards	Corrective Action
624	once/12 hr CL: 20 µg/L	(Appendix 1) Response range for each compound	Supelco Accu-standard prepared internally	Reanalyze. Re-run a new initial calibration if continuing calibration (CC) fails.
625	once/12 hr CL: Acid - 100 µg/L BN - 50 µg/L	%Difference (D) < 20% (Appendix 2) for all target compounds except 4	Supelco, Accu-standard prepared internally	If CC fails reinject appropriate level standards to complete initial calibration.
8240	once/12 hr CL: 50 µg/L	%D < 25% for CCC (Appendix 4) Minimum RF = 0.300 for SPCC (0.250 for bromoform)	Supelco Accu-standard prepared internally	Re-tune, check purge flow, change trap. Re-analyze. If CC fails, rerun initial calibration.
8270	once/12 hr CL: 50 µg/L	%D < 25% for CCC (Appendix 3) for all target compounds, Minimum RF = 0.050 for SPCC	Supelco Accu-standard prepared internally	Re-tune and rerun if CC fails, then rerun initial calibration.
CLP 3/90 VOA	once/12 hr CL: 50 µg/L	%D < or = 25% for most compounds (Appendix 5)	Restek	Re-tune, check purge flow, change trap. Re-analyze. If CC fails, rerun initial calibration.
CLP 3/90 SV	once/12 hr CL: 20 µg/L	(Appendix 5) All maximum %D and minimum RRF criteria must be met.	Restek	Re-tune. Reanalyze. If CC fails, rerun initial calibration.
10/92 SAM LC VOA	once/12 hr CL: Ketones: 25 µg/L All others: 5 µg/L	%D < or = 25% for most compounds.	Restek	Re-tune, check purge flow, change trap. Re-analyze. If If CC fails, rerun initial calibration.
10/92 SAM LC SV	once/12 hr CL: 20 µg/L	(Appendix 6) All maximum %D and minimum RRF criteria must be met.	Restek	Re-tune and re-analyze. If CC fails, rerun initial calibration.

Appendix #1: Method 624

Compound	12-Hour Check Standard for 20 µg/L 624 Standard (µg/L)
chloromethane	D - 40.8
bromomethane	2.8 - 37.2
chloroethane	0.8 - 38.2
methylene chloride	12.1 - 27.9
1,1-dichloroethene	10.1 - 28.9
1,1-dichloroethane	14.5 - 25.5
1,2-dichloroethene (total)	13.8 - 26.1
chloroform	13.5 - 26.5
1,2-dichloroethane	13.6 - 26.4
1,1,1-trichloroethane	15.0 - 25.0
carbon tetrachloride	14.6 - 25.4
bromodichloromethane	13.1 - 26.9
1,2-dichloropropane	6.8 - 33.2
trans-1,3-dichloropropene	10.0 - 30.0
trichloroethene	13.3 - 26.7
chlorodibromomethane	13.5 - 26.5
1,1,2-trichloroethane	14.2 - 25.0
benzene	12.8 - 27.2
cis-1,3-dichloropropene	4.8 - 35.2
2-chloroethyl vinyl ether	D - 44.8
bromoform	14.2 - 25.8
tetrachloroethene	14.7 - 25.3
1,1,2,2-tetrachloroethane	12.1 - 27.9
toluene	14.8 - 25.1
chlorobenzene	13.2 - 26.8
ethylbenzene	11.8 - 28.2

Appendix #2: Method 625

Exceptions to criteria set forth in GC/MS Method 625 calibration table:

The following four compounds need not meet < 20% D (difference between computed and expected recoveries) in the continuing calibration (12-hour check standard):

N-nitrosodimethylamine
hexachlorocyclopentadiene
diphenylamine (N-nitroso)
benzidine

Appendix #3: Method 8270

Calibration Check Compounds
The following compounds must meet a maximum of 30% RSD in the initial calibration and a maximum of 25% difference in the continuing calibration (12-hour check standard):

1,4-dichlorobenzene
phenol
2-nitrophenol
2,4-dichlorophenol
hexachlorobutadiene
P-chloro-M-cresol
2,4,6-trichlorophenol
acenaphthene
N-nitrosodiphenylamine
pentachlorophenol
fluoroanthene
Di-N-octyl phthalate
benzo(a)pyrene

System Performance Check Compounds
The following compounds must have a minimum average relative response factor of 0.050 in the initial calibration and a minimum relative response factor of 0.050 in the continuing calibration (12-hour check standard):

N-nitroso-Di-N-propylamine	2,4-dinitrophenol
hexachlorocyclopentadiene	4-nitrophenol

Appendix #4: Method 8240

Calibration Check Compounds
The following compounds must meet a maximum of 30% RSD in the initial calibration and a maximum of 25% difference in the continuing calibration (12-hour check standard):

vinyl chloride	1,2-dichloropropane
1,1-dichloroethene	toluene
chloroform	ethylbenzene

System Performance Check Compounds
The following compounds must have a minimum average relative response factor of 0.300 in the initial calibration and a minimum relative response factor of 0.300 in the initial calibration and a minimum relative response factor of 0.300 (0.250 for bromoform) in the continuing calibration (12-hour check standard):

chloromethane	1,1-dichloroethane
chlorobenzene	bromoform
1,1,2,2-tetrachloroethane	

Appendix #5: Table 8-1A. Relative Response Factor Criteria for Initial and Continuing Calibration of Volatile Organic Compounds (CLP 3/90, Volatile Organics)

Volatile Compound	Minimum RRF	Maximum %RSD	Maximum %D
bromomethane	0.100	20.5	25.0
vinyl chloride	0.100	20.5	25.0
1,1-dichloroethene	0.100	20.5	25.0
1,1-dichloroethane	0.200	20.5	25.0
chloroform	0.200	20.5	25.0
1,2-dichloroethane	0.100	20.5	25.0
1,1,1-trichloroethane	0.100	20.5	25.0
carbon tetrachloride	0.100	20.5	25.0
bromodichloromethane	0.200	20.5	25.0
cis-1,3-dichloropropene	0.200	20.5	25.0
trichloroethene	0.300	20.5	25.0
dibromochloromethane	0.100	20.5	25.0
1,1,2-trichloroethane	0.100	20.5	25.0
benzene	0.500	20.5	25.0
trans-1,3-dichloropropene	0.100	20.5	25.0
bromoform	0.100	20.5	25.0
tetrachloroethene	0.200	20.5	25.0
1,1,2,2-tetrachloroethane	0.500	20.5	25.0
toluene	0.400	20.5	25.0
chlorobenzene	0.500	20.5	25.0
ethylbenzene	0.100	20.5	25.0
styrene	0.300	20.5	25.0
xylenes (total)	0.300	20.5	25.0
bromofluorobenzene	0.200	20.5	25.0

Because performance data indicate erratic and poor linearity, the following compounds have no maximum %RSD or maximum %D criteria. However, these compounds must meet a minimum RRF criterion of 0.010:

acetone	1,2-dichloropropane
2-butanone	2-hexanone
carbon disulfide	methylene chloride
chloroethane	4-methyl-2-pentanone
chloromethane	toluene-d_8
1,2-dichloroethene (total)	1,2-dichloroethane-d_4

The response factors of the compounds listed in Table 8-1A must meet the minimum RRF criteria at each concentration level and maximum %RSD criteria for the initial calibration, with allowance made for up to two volatile target compounds. However, the RRFs for those two compounds must be ≥ 0.010, and the %RSD of those two compounds must be $\leq 40.0\%$ for the initial calibration to be acceptable.

Appendix #5 (CONTINUED): Table 8-2A. Relative Response Factor Criteria for Initial and Continuing Calibration of Semivolatile Organic Compounds (CLP 3/90, Semivolatile Organics)

Semivolatile Compound	Minimum RRF	Maximum %RSD	Maximum %D
phenol	0.800	20.5	25.0
bis(-2-chloroethyl)ether	0.700	20.5	25.0
2-chlorophenol	0.800	20.5	25.0
1,3-dichlorobenzene	0.600	20.5	25.0
1,4-dichlorobenzene	0.500	20.5	25.0
1,2-dichlorobenzene	0.400	20.5	25.0
2-methylphenol	0.700	20.5	25.0
4-methylphenol	0.600	20.5	25.0
N-nitroso-di-n-propylamine	0.500	20.5	25.0
hexachloroethane	0.300	20.5	25.0
nitrobenzene	0.200	20.5	25.0
isophorone	0.400	20.5	25.0
2-nitrophenol	0.100	20.5	25.0
2,4-dimethylphenol	0.200	20.5	25.0
bis(-2-chloroethoxy)methane	0.300	20.5	25.0
2,4-dichlorophenol	0.200	20.5	25.0
1,2,4-trichlorobenzene	0.200	20.5	25.0
naphthalene	0.700	20.5	25.0
4-chloro-3-methylphenol	0.200	20.5	25.0
2-methylnaphthalene	0.400	20.5	25.0
2,4,6-trichlorophenol	0.200	20.5	25.0
2,4,5-trichlorophenol	0.200	20.5	25.0
2-chloronaphthalene	0.800	20.5	25.0
acenaphthylene	1.300	20.5	25.0
2,6-dinitrotoluene	0.200	20.5	25.0
acenaphthene	0.800	20.5	25.0
dibenzofuran	0.800	20.5	25.0
2,4-dinitrotoluene	0.200	20.5	25.0
4-chlorophenol-phenylether	0.400	20.5	25.0
fluorene	0.900	20.5	25.0
4-bromophenyl-phenylether	0.100	20.5	25.0
hexachlorobenzene	0.100	20.5	25.0
pentachlorophenol	0.050	20.5	25.0
phenanthrene	0.700	20.5	25.0
anthracene	0.700	20.5	25.0
fluoroanthene	0.600	20.5	25.0
pyrene	0.600	20.5	25.0
benzo(a)anthracene	0.800	20.5	25.0
chrysene	0.700	20.5	25.0
benzo(b)fluoranthene	0.700	20.5	25.0
benzo(k)fluoranthene	0.700	20.5	25.0
benzo(a)pyrene	0.700	20.5	25.0
indeno(1,2,3-cd)pyrene	0.500	20.5	25.0

Appendix #5 (CONTINUED): Table 8-2A. Relative Response Factor Criteria for Initial and Continuing Calibration of Semivolatile Organic Compounds (CLP 3/90, Semivolatile Organics)

Semivolatile Compound	Minimum RRF	Maximum %RSD	Maximum %D
dibenzo(a,h)anthracene	0.400	20.5	25.0
benzo(g,h,i)perylene	0.500	20.5	25.0
nitrobenzene-d_5	0.200	20.5	25.0
2-fluorobiphenyl	0.700	20.5	25.0
terphenyl-d_{14}	0.500	20.5	25.0
phenol-d_5	0.800	20.5	25.0
2-fluorophenol	0.600	20.5	25.0
2-chlorophenol-d_4	0.800	20.5	25.0
1,2-dichlorobenzene-d_4	0.400	20.5	25.0

The following compounds have no maximum %RSD or maximum %D criteria. However, these compounds must meet a minimum RRF criterion of 0.010:

2,2'-oxybis(1-chloropropane)
4-chloroaniline
hexachlorobutadiene
hexachlorocyclopentadiene
2-nitroaniline
dimethylphthalate
3-nitroaniline
2,4-dinitrophenol
1-nitrophenol
diethylphthalate
4-nitroaniline
4,6-dinitro-2-methylphenol
N-nitrosodiphenylamine
Di-N-butylphthalate
butylbenzylphthalate
3,3'-dichlorobenzidine
bis(2-ethylhexyl)phthalate
Di-N-octylphthalate
2,4,5-tribromophenol
carbazole

The response factors of the compounds listed in Table 8-2A must meet the minimum RRF criteria at each concentration level and maximum %RSD criteria for the initial calibration, with allowance made for up to four semivolatile target compounds. However, the RRFs for those four compounds must be > 0.010, and the %RSD of those four compounds must be $\leq$ 40.0% for the initial calibration to be acceptable.

Appendix #6: Table 8-3A. Technical Acceptance Criteria for Initial and Continuing Calibration of Volatile Organic Compounds (10/92 SAM, Low Concentration VOA Organics)

Volatile Compound	Minimum RRF	Maximum %RSD	Maximum %D
benzene	0.500	30.0	± 30.0
bromochloromethane	0.05	30.0	± 30.0
bromodichloromethane	0.200	30.0	± 30.0
bromoform	0.05	30.0	± 30.0
bromomethane	0.100	30.0	± 30.0
carbon tetrachloride	0.100	30.0	± 30.0
chlorobenzene	0.500	30.0	± 30.0
chloroform	0.200	30.0	± 30.0
dibromochloromethane	0.100	30.0	± 30.0
1,2-dibromoethane	0.100	30.0	± 30.0
1,2-dichlorobenzene	0.400	30.0	± 30.0
1,3-dichlorobenzene	0.600	30.0	± 30.0
1,4-dichlorobenzene	0.500	30.0	± 30.0
1,1-dichloroethane	0.200	30.0	± 30.0
1,2-dichloroethane	0.100	30.0	± 30.0
1,1-dichloroethene	0.100	30.0	± 30.0
cis-1,3-dichloropropene	0.200	30.0	± 30.0
trans-1,3-dichloropropene	0.100	30.0	± 30.0
ethylbenzene	0.100	30.0	± 30.0
styrene	0.300	30.0	± 30.0
1,1,2,2-tetrachloroethane	0.100	30.0	± 30.0
tetrachloroethene	0.200	30.0	± 30.0
toluene	0.400	30.0	± 30.0
1,1,1-trichloroethane	0.100	30.0	± 30.0
1,1,2-trichloroethane	0.100	30.0	± 30.0
trichloroethene	0.300	30.0	± 30.0
vinyl chloride	0.100	30.0	± 30.0
xylenes (total)	0.300	30.0	± 30.0
4-bromofluorobenzene	0.200	30.0	± 30.0

The following compounds have no maximum %RSD or maximum %D criteria, but must meet a minimum RRF criteria of 0.010:

carbon disulfide
chloroethane
chloromethane
cis-1,2-dichloroethene
trans-1,2-dichloroethene
1,2-dichloropropane
methylene chloride

NOTE: Presently, the U.S. EPA has set no minimum RRF or %RSD criteria for acetone, 2-butanone, 1,2-dibromo-3-chloropropane, 2-hexanone, or 4-methyl-2-pentanone.

Appendix #6 (CONTINUED): Table 8-3A. Technical Acceptance Criteria for Initial and Continuing Calibration of Semivolatile Organic Compounds (10/92 SAM, Low Concentration Semivolatile Organics)

Semivolatile Compound	Minimum RRF	Maximum %RSD	Maximum %D
phenol	0.800	20.5	± 25.0
bis(-2-chloroethyl)ether	0.700	20.5	± 25.0
2-chlorophenol	0.700	20.5	± 25.0
2-methylphenol	0.700	20.5	± 25.0
4-methylphenol	0.600	20.5	± 25.0
n-nitroso-di-n-propylamine	0.500	20.5	± 25.0
hexachloroethane	0.300	20.5	± 25.0
nitrobenzene	0.200	20.5	± 25.0
isophorone	0.400	20.5	± 25.0
2-nitrophenol	0.100	20.5	± 30.0
2,4-dimethylphenol	0.200	20.5	± 30.0
1,2,4-trichlorobenzene	0.200	20.5	± 25.0
naphthalene	0.700	20.5	± 25.0
4-chloro-3-methylphenol	0.200	20.5	± 25.0
2-methylnaphthalene	0.400	20.5	± 25.0
2,4,6-trichlorophenol	0.200	20.5	± 25.0
2,4,5-trichlorophenol	0.200	20.5	± 25.0
2-chloronaphthalene	0.800	20.5	± 25.0
acenaphthylene	1.300	20.5	± 25.0
acenaphthene	0.800	20.5	± 25.0
dibenzofuran	0.800	20.5	± 25.0
2,4-dinitrotoluene	0.200	20.5	± 30.0
2,6-dinitrotoluene	0.200	20.5	± 25.0
4-chlorophenol-phenylether	0.400	20.5	± 25.0
fluorene	0.900	20.5	± 25.0
4-bromophenyl-phenylether	0.100	20.5	± 25.0
hexachlorobenzene	0.100	20.5	± 25.0
pentachlorophenol	0.050	20.5	± 25.0
phenanthrene	0.700	20.5	± 25.0
anthracene	0.700	20.5	± 25.0
fluoranthene	0.600	20.5	± 25.0
pyrene	0.600	20.5	± 25.0
benzo(a)anthracene	0.800	20.5	± 25.0
chrysene	0.700	20.5	± 25.0
benzo(b)fluoranthene	0.700	20.5	± 25.0
benzo(k)fluoranthene	0.700	20.5	± 25.0
benzo(a)pyrene	0.700	20.5	± 25.0
indeno(1,2,3-cd)pyrene	0.500	20.5	± 25.0
dibenzo(a,h)anthracene	0.400	20.5	± 25.0

Appendix #6 (CONTINUED): Table 8-3A. Technical Acceptance Criteria for Initial and Continuing Calibration of Semivolatile Organic Compounds (10/92 SAM, Low Concentration Semivolatile Organics)

Semivolatile Compound	Minimum RRF	Maximum %RSD	Maximum %D
benzo(g,h,i)peryiene	0.500	20.5	± 25.0
phenol-d_5	0.800	20.5	± 25.0
2-fluorophenol	0.600	20.5	± 25.0
terphenyl-d_{14}	0.500	20.5	± 25.0
2-fluorobiphenyl	0.700	20.5	± 25.0

The following compounds have no maximum %RSD or maximum %D criteria. However, these compounds must meet a minimum RRF criterion of 0.010:

2,2'-oxybis(1-chloropropane)
4-chloroaniline
hexachlorobutadiene
hexachlorocyclopentadiene
2-nitroaniline
dimethylphthalate
3-nitroaniline
2,4-dinitrophenol
4-nitrophenol
diethylphthalate
4-nitroaniline
4,6-dinitro-2-methylphenol
n-nitrosodiphenylamine
Di-n-butylphthalate
butylbenzylphthalate
3,3'-dichlorobenzidine
bis(2-ethylhexyl)phthalate
Di-n-octylphthalate
2,4,6-tribromophenol
nitrobenzene-d_5

Table 8-7. Initial Calibration Procedures for Analytical Equipment in the GC Lab

Method	No. of Stds. for Initial Calibration	Calibration Frequency	Acceptance Criteria
601	3	When continuing calibration fails	< 25% RSD for all compounds. Use average RF if %RSD < 10%. Otherwise, use calibration curve.
602	3	When continuing calibration fails.	< 25% RSD for all compounds. Use average RF if %RSD < 10%. Otherwise, use calibration curve.
608	15	Every 72 hours or when calibration check standards fail.	Linearity standards must be <10% RSD and DBC drift must be < 2%. DDT and Endrin degradation must be < 20% combined, and %RT drift must be < 2% for packed column or < 1.5% for capillary column.
8010	5	When continuing calibration fails.	< 25% RSD for all compounds. Use average RF if %RSD < 20%. Otherwise use calibration curve.
8020	5	When continuing calibration fails.	< 25% RSD for all compounds. Use average RF if %RSD < 20%. Otherwise use calibration curve.
8080	15	When calibration check standards fail.	Linearity standards must be ≤ 20% RSD. DDT or Endrin degradation must be ≤ 20%. Surrogate %RT drift must be < 2% for packed column or <1.5% for capillary column.
8150	5	When calibration check standards fail.	≤ 20% RSD for all compounds. ≤ 1.5% RT drift from high level initial standard.
8140	5	When calibration check standards fail.	≤ 20% RSD for all compounds. ≤ 1.5% RT drift from initial standard.

Table 8-7 (CONTINUED). Initial Calibration Procedures for Analytical Equipment in the GC Lab

Method	No. of Stds. for Initial Calibration	Calibration Frequency	Acceptance Criteria
CLP 3/90, 10/92 SAM	17	When continuing calibration fails.	%RSD ≤ 20% for TCL except up to two may be ≤ 30%. %RSD < 30% for TCX and DBC. **Resolution:** RESCK ≥ 60% PEM = 100% INDAM ≥ 90% INDBM ≥ 90% %D of PEM ±25% **RT windows:** established from mean RT of three-point ± 0.05 min for compounds that elute before heptachlor epoxide, ± 0.07 min for other compounds except ± 0.10 min for DCB. **Instrument blank:** TCL compounds < 1/2 CRQL of water.

Table 8-8. Continuing Calibration Procedures for Analytical Equipment in the GC Lab

Method	Frequency of Continuing Calibration	Acceptance Criteria	Sources for Standards	Corrective Action
601	once/24 hr	All compounds within acceptance limits (Appendices 7 and 8)	Supelco Accu-Standard	Reanalyze standard. If unacceptable, repeat initial calibration.
602	once/24 hr	All compounds within acceptance limits (Appendices 7 and 8)	Supelco Accu-Standard	Reanalyze standard. If unacceptable, repeat initial calibration.
608/8080 Pesticide/PCBs	after every tenth sample in sequence	Response factor (RFs) must be ± 15%D from the initial calibration RFs. DDT and Endrin degradation must be ≤ 20%. Surrogate %RT drift must be < 2% (packed column), and < 1.5% (capillary column).	Prepared internally from neat (pure) materials	Septum change and column maintenance necessary. Rerun initial calibration if reanalysis fails.
CLP Pesticides/ PCBs	once/12 hr	RFs must be ± 15%D from initial calibration RFs. DDT and Endrin degradation must be ≤ 20% each PEM, but ≤ 30% combined. Surrogate %RT drift must be < 2% (packed column), or < 1.5% (capillary column).	Restek	Change septum and perform column maintenance. Rerun initial calibration if reanalysis fails.
8010	after every tenth sample in sequence	All compounds within acceptance limits. (Appendices 9 and 10)	Supelco, Accu-Standard	Reanalyze standard. If unacceptable, repeat initial calibration.
8020	after every tenth sample in sequence	All compounds within acceptance limits. (Appendices 9 and 10)	Supelco, Accu-Standard	Reanalyze standard. If unacceptable, repeat initial calibration.

Table 8-8 (CONTINUED). Continuing Calibration Procedures for Analytical Equipment in the GC Lab

Method	Frequency of Continuing Calibration	Acceptance Criteria	Sources for Standards	Corrective Action
8150	once/ 10 samples	RF %D ± 15% for all compounds. RT drift ≤ 1.5% from initial high level standard.	Prepared internally	Change septum and perform column maintenance. Rerun initial calibration if reanalysis fails.
8140	once/ 10 samples	RF %D ± 15% for all compounds. RT drift ≤ 1.5% from initial high level standard.	Prepared internally	Change septum and perform column maintenance. Rerun initial calibration if reanalysis fails.

Appendix #7: Table 8-4A Standard Concentration and Check Standard Acceptance Range for Method 601

Compound	Concentration of Standard (µg/L)	Check Standard Acceptance Range (µg/L)
chloromethane	20	11.9 - 28.1
vinyl chloride	20	13.7 - 26.3
bromomethane	20	11.7 - 28.3
chloroethane	20	15.4 - 24.6
1,1-dichloroethane	20	12.6 - 27.4
methylene chloride	20	15.5 - 24.5
t-1,2-dichloroethene	20	12.8 - 27.2
1,1-dichloroethane	20	16.8 - 23.2
chloroform	20	15.0 - 25.0
1,1,1-trichloroethane	20	14.2 - 25.8
carbon tetrachloride	20	13.7 - 26.3
1,2-dichloroethane	20	14.3 - 25.7
trichloroethene	20	15.4 - 24.6
1,2-dichloropropane	20	14.8 - 25.2
bromodichloromethane	20	15.2 - 24.8
2-chloroethyl vinyl ether	20	12.0 - 28.0
c-1,3-dichloropropene	20	12.8 - 27.2
t-1,3-dichloropropene	20	12.8 - 27.2
1,1,2-trichloroethane	20	15.7 - 24.3
tetrachloroethene	20	14.0 - 26.0
dibromochloromethane	20	13.1 - 26.9
chlorobenzene	20	14.4 - 25.6
bromoform	20	14.7 - 25.3
1,1,2,2-tetrachloroethane	20	8.8 - 30.2
1,3-dichlorobenzene	20	9.9 - 30.1
1,4-dichlorobenzene	20	13.9 - 26.1
1,2-dichlorobenzene	20	14.0 - 26.0

Appendix #8: Table 8-5A. Standard Concentration and Check Standard Acceptance Range for Method 602

Compound	Concentration of Standard (µg/L)	Check Standard Acceptance Range (µg/L)
benzene	20	15.4 - 24.6
toluene	20	15.5 - 24.5
chlorobenzene	20	16.1 - 23.9
ethylbenzene	20	12.6 - 27.4
1,3-dimethylbenzene	20	12.6 - 27.4
1,4-dimethybenzene	20	12.6 - 27.4
1,2-dimethylbenzene	20	12.6 - 27.4
styrene	20	16.1 - 23.9
1,3-dichlorobenzene	20	14.5 - 25.5
1,4-dichlorobenzene	20	13.8 - 26.1
1,2-dichlorobenzene	20	13.6 - 26.4

Appendix #9: Table 8-6A. Standard Concentration and Check Standard Acceptance Range for Method 8010

Compound	Concentration of Standard (µg/L)	Check Standard Acceptance Range (µg/L)
chloromethane	20	11.9 - 28.1
vinyl chloride	20	13.7 - 26.3
bromomethane	20	11.7 - 28.3
chloroethane	20	15.4 - 24.6
1,1-dichloroethane	20	12.6 - 27.4
methylene chloride	20	15.5 - 24.5
t-1,2-dichloroethene	20	12.8 - 27.2
1,1-dichloroethane	20	16.8 - 23.2
chloroform	20	15.0 - 25.0
1,1,1-trichloroethane	20	14.2 - 25.8
carbon tetrachloride	20	13.7 - 26.3
1,2-dichloroethane	20	14.3 - 25.7
trichloroethene	20	15.4 - 24.6
1,2-dichloropropane	20	14.8 - 25.2
bromodichloromethane	20	15.2 - 24.8
2-chloroethyl vinyl ether	20	12.0 - 28.0
c-1,3-dichloropropene	20	12.8 - 27.2
t-1,3-dichloropropene	20	12.8 - 27.2
1,1,2-trichloroethane	20	15.7 - 24.3
tetrachloroethene	20	14.0 - 26.0
dibromochloromethane	20	13.1 - 26.9
chlorobenzene	20	14.4 - 25.6
bromoform	20	14.7 - 25.3
1,1,2,2-tetrachloroethane	20	8.8 - 30.2
1,3-dichlorobenzene	20	9.9 - 30.1
1,4-dichlorobenzene	20	13.9 - 26.1
1,2-dichlorobenzene	20	14.0 - 26.0

Appendix #10: Table 8-7A. Standard Concentration and Check Standard Acceptance Range for Method 8020

Compound	Concentration of Standard (µg/L)	Check Standard Acceptance Range (µg/L)
methyl-t-butyl ether	60	16.8 - 100.2
benzene	20	15.4 - 24.6
toluene	20	15.5 - 24.5
chlorobenzene	20	16.1 - 23.9
ethylbenzene	20	12.6 - 27.4
1,4-dimethylbenzene	20	12.6 - 27.4
1,3-dimethylbenzene	20	12.6 - 27.4
1,2-dimethylbenzene	20	12.6 - 27.4
styrene	20	16.1 - 23.9
1,3-dichlorobenzene	20	14.5 - 25.5
1,4-dichlorobenzene	20	13.9 - 26.1
1,2-dichlorobenzene	20	13.6 - 26.4

Table 8-9. Initial Calibration Procedures for Analytical Equipment in the Inorganics Lab

Method	No. Stds. for Initial Calibration	Calibration Frequency and Concentration Range	Method of Curve Generation	Acceptance Criteria
ICP Metals	5	twice daily/every 10-hr shift (0-100 ppm)	two-point curve with automatic instrument software calculation.	ICV ± 10% of true value
GFAAS Metals	4	daily/with each use (0-60 ppb)	linear regression, automatic instrument software calculation	ICV ± 10% of true value
Mercury by CVAAS	6	daily/with each use (0-10 ppb)	linear regression	correlation coefficient > 0.995
Cyanide	6	daily/with each use (0-300 ppb)	linear regression	correlation coefficient > 0.995
Phenol	7	with each use (0-300 ppb)	linear regression	correlation coefficient > 0.995
Fluoride	7	with each use (0-5 ppm)	linear regression; each chord is separately calculated and reported.	total curve correlation coefficient > 0.995
Alkalinity	7	with each use (0-500 ppm)	linear regression; each chord is separately calculated and reported	total curve correlation coefficient > 0.995
Hexavalent Chromium	7	with each use (0-400 ppb)	linear regression; each chord is separately calculated and reported	total curve correlation coefficient > 0.995
Ammonia	8	with each use (0-20 pm)	linear regression; each chord is separately calculated and reported	total curve correlation coefficient > 0.995

Table 8-9 (CONTINUED). Initial Calibration Procedures for Analytical Equipment in the Inorganics Lab

Method	No. Stds. for Initial Calibration	Calibration Frequency and Concentration Range	Method of Curve Generation	Acceptance Criteria
Sulfates	4	with each use (0 - 40 ppm)	linear regression	correlation coefficient > 0.995
Chloride	10	with each use (0 - 300 ppm)	linear regression, each chord is separately calculated and reported	total curve correlation coefficient > 0.995
Hardness	5	with each use (0 - 50 ppm)	linear regression, each chord is separately calculated and reported	total curve correlation coefficient > 0.995
Nitrate	8	with each use (0 - 2000 ppb)	linear regression	correlation coefficient > 0.995
Nitrite	8	with each use (0 - 2000 ppb)	linear regression	correlation coefficient > 0.995

Table 8-10. Continuing Calibration Procedures for Analytical Equipment in the Inorganics Lab

Method	Frequency and Concentration of Continuing Calibration	Acceptance Criteria	Sources for Standards	Corrective Action
ICP Metals 200.7 6010 CLP 3/90	10% or every two hours, whichever is more frequent (500 ppb - 5000 ppb, concentration varies for individual analytes within a calibration standard)	± 10% true ± 5% true for 200.7	Spex	Halt analyses and recalibrate and reanalyze previous 10 samples.
GFAAS Metals As: 206.2, 7060, CLP Pb: 238.2, 7421, CLP Se: 270.2, 7740, CLP Tl: 278.2, 7841, CLP	10% or every two hours, whichever is more frequent (30 ppb)	± 10% true	Spex Inorganic Ventures	Halt analyses and recalibrate and reanalyze previous 10 samples.
Mercury by CVAAS 245.1, 7470, CLP 3/90, 7471, 245.5	10% or every two hours, whichever is more frequent (3 ppb)	± 20% true ± 10% true for 245.1/245.5	Baker/EPA	Halt analyses and recalibrate and reanalyze previous 10 samples.
Cyanide 335.2-.3, CLP 3/90	10% or every two hours, whichever is more frequent (100 ppb)	± 15% true	Fisher/EPA	Rerun all samples not preceded and/or followed by acceptable ICV/ICB and CCV/CCB.
Phenol 420.1-.2, 9066	10% or every two hours, whichever is more frequent (100 ppb)	± 15% true	Fisher	Rerun all samples not preceded and/or followed by acceptable ICV/ICB and CCV/CCB.

Table 8-10 (CONTINUED). Continuing Calibration Procedures for Analytical Equipment in the Inorganics Lab

Method	Frequency and Concentration of Continuing Calibration	Acceptance Criteria	Sources for Standards	Corrective Action
Fluoride 340.2, 10-109-12-2-A	10% (2 ppm)	± 15% true	Fisher	Rerun all samples not preceded and/or followed by acceptable ICV/ICB and CCV/CCB.
Sulfate 375.4, 9038	10% (20 ppm)	± 15% true	Fisher	Rerun all samples not preceded and/or followed by acceptable ICV/ICB and CCV/CCB.
Chloride 325.2, 9251, 10-117-07-1-A	10% (240 ppm)	± 15% true	Fisher	Rerun all samples not preceded and/or followed by acceptable ICV/ICB and CCV/CCB.
Alkalinity 310.2, 10-303-31-1-A	10% (300 ppm)	± 15% true	Baker	Rerun all samples not preceded and/or followed by acceptable ICV/ICB and CCV/CCB.
Hexavalent Chromium 10-124-13-1-A	10% (200 ppb)	± 15% true	Fisher	Rerun all samples not preceded and/or followed by acceptable ICV/ICB and CCV/CCB.
Ammonia 350.2, 10-107-06-1-A	10% (8 ppm)	± 15% true	Fisher	Rerun all samples not preceded and/or followed by acceptable ICV/ICB and CCV/CCB.
Hardness 130.1, 10-301-31-1-A	10% (20 ppm)	± 15% true	Fisher	Rerun all samples not preceded and/or followed by acceptable ICV/ICB and CCV/CCB.

Table 8-10 (CONTINUED). Continuing Calibration Procedures for Analytical Equipment in the Inorganics Lab

Method	Frequency and Concentration of Continuing Calibration	Acceptance Criteria	Sources for Standards	Corrective Action
Nitrate 353.2	10% (1000 ppb)	± 15% true	Fisher	Rerun all samples not preceded and/or followed by acceptable ICV/ICB and CCV/CCB.
Nitrite 353.2	10% (1000 ppb)	± 15% true	Fisher	Rerun all samples not preceded and/or followed by acceptable ICV/ICB and CCV/CCB.

Table 8.11. Initial Calibration Procedures for Analytical Equipment in the Organic Characterization Lab

Method	No. of Stds. for Initial Calibration	Calibration Frequency and Concentration Range	Acceptance Criteria	Corrective Action
Total Petroleum Hydrocarbons 9071, 503E, 418.1	6	daily/with each use (0 - 50 ppm)	correlation coefficient must be $\geq$ 0.995	Rerun calibration if it fails.
Oil and Grease 503B, 9071	6	daily/with each use (0 - 50 ppm)	correlation coefficient must be $\geq$ 0.995	Rerun calibration if it fails.
Total Organic Carbon 505	2	daily/with each use (0 - 50 ppm)	± 5%D for two injections	Rerun calibration if it fails.
Total Organic Halides	1	trichlorophenol (TCP) with each use (100 ppm)	± 20% of true	Rerun calibration if it fails.

Table 8-11 (CONTINUED). Continuing Calibration Procedures for Analytical Equipment in the Organic Characterization Lab

Method	Frequency and Concentration of Continuing Calibration	Acceptance Criteria	Sources for Standards	Corrective Action
Total Petroleum Hydrocarbons 9071, 503E, 418.1	once / 10 analyses (25 ppm)	0 std. ± 1 ppm 25 std. ± 5 ppm	Fisher	If CC fails, rerun samples run before CC failed, check CCV.
Oil and Grease 503B, 9071	once / 10 analyses (25 ppm)	0 std. ± 1 ppm 25 std. ± 5 ppm	Fisher	Rerun calibration if it fails/ If CC fails, rerun samples run before CC failed, check CCV.
Total Organic Carbon 505	once / 10 analyses (50 ppm)	± 5%	potassium hydrogen phthalate from Aldrich Chemical Co.	If CCV failed, rerun 10 preceding samples if calibration fails, check CCV.
Total Organic Halides 506/9020	once / 10 analyses (50 ppm)	± 25%	2,4,6-trichlorophenol from Aldrich Chemical Co.	Polish titration cell electrodes. Flush titration cell with 70% acetic acid. Recoat electrodes, recalibrate, and reanalyze samples.

8-12. Continuing Calibration Procedures for Analytical Equipment in the Radiochemistry Lab

Method	Frequency of Continuing Calibration	Acceptance Criteria	Sources for Standards	Corrective Action
Gross Alpha/Beta 9310	daily	± 3 sigma of true value	NIST	Rerun calibration verification and contact manager/service representative after second failure.
Radium-226 903.1 (A)	annually by manufacturer	NA	NIST	Return to manufacturer as required.
Total Radium 9315	daily	± 3 sigma of true value	NIST	Rerun calibration if it fails.
Uranium-02 Isotopic U-234/238	once monthly/ as used	within 40 KeV of correct energy	NIST	Rerun calibration verification. Contact manager/service representative after second failure.

8.2 Standards Traceability

All standards used for calibration are either provided by the EPA ("EPA-certified"), traceable to an EPA or NIST standard source, or traceable by statistical comparison with an independently-prepared standard source. The latter method is described fully in the 3/90 CLP SOW (3/90 SOW).

Organic standards used in the GC and GC/MS Laboratories are prepared by a full-time organic standards chemist. Standards used for the CLP 3/90 SOW are purchased in ampulated form from a commercial vendor. The vendor provides a certification package which establishes statistical traceability.

Every compound is analyzed for purity and identity by refractive index/melting point (RI/MP), GC/FID, and GC/MS (using high resolution capillary columns). Pesticides are analyzed by GC/ECD and the volatile gases are analyzed by GC/ELCD.In addition to the RI/MP values and analytical chromatograms, the certification data package includes the GC/MS spectra, purity data, gravimetric records, and statistical comparisons of independent solutions.

All standards are prepared from neat materials of 97% purity or higher. (Most are greater than 99% pure.) Some isomers are purchased in mixtures and each lot may vary in composition of the individual isomers (e.g., cis- and trans-1,3-dichloropropene), which must be taken into consideration in quantitating unknowns containing these isomers.

Commercially prepared standards require only dilution to the working level concentration. The accuracy of the dilution is checked by comparing the new working level standard lot concentrations against the previously prepared lot (before expiration). This standard lot preparation test is described fully in a GC/MS Laboratory SOP. The QA department performs a quarterly audit of new standard lot tests.

Standards used for non-CLP 3/90 SOW analyses are prepared from neat chemicals of guaranteed purity. The stock (primary) standard is then diluted and the working level standard is tested as described above. Standards are assigned an ID number and a lot number. The ID number refers to the "recipe" used in the preparation, the requirements for labeling the standard bottle, the solvent(s) used, and the expiration period. The lot number is a five-digit sequentially assigned number that refers to the particular preparation of a standard. The preparation may be tracked to the standard preparation logbook through this lot number. The chemist records the weights, volumes, concentrations, and vendor reference codes of stock of intermediate standards used; the solvent (including vendor, grade, and vendor lot number); his or her own initials; and the date of preparation. The vendor reference codes are cross-referenced to a separate inventory logbook that catalogs all neat or stock standards received by the laboratory, the date of receipt, vendor, and standard purity. New volatile stock standard lots are prepared every week. Working level volatile standards are prepared from these stocks each week.

Semivolatile standard stocks are prepared every seven weeks, and working level standards are prepared every four to six weeks, depending on the laboratory consumption rate. Fresh volatile stock standards are prepared monthly by the organic standards chemist and stored frozen in individual mininerts in the Standards Laboratory. Each week the chemist prepares and issues working level standards to the laboratory. Fresh semivolatile stock standards are prepared every six months. Working level standards are prepared every six weeks or as often

as needed.

Pesticide standard stocks are prepared yearly, and working level standards are prepared every two to four weeks, depending on the standard types (calibration, PEM mixture, surrogates, etc.) and the rate at which the laboratory consumes the standards. Aroclor standard stocks are also prepared yearly, with working level standards prepared every 2-3 months in most cases, not to exceed six months. In all instances, standard expiration periods are shorter than or equal to those specified by the CLP 3/90 SOW.

All volatile standards are stored in a freezer at -10 to -20°C, in a separate refrigeration unit. Standards are stored separately from samples in all cases. Standards used in non-GC/MS and GC laboratories are prepared by the chemists assigned to these areas. Trace metals standards are purchased from commercial vendors and diluted and certified internally as described above for GC/MS and GC standards. Standards from a second source (different vendor or different stock lot from the same vendor) are used to verify the stock traceability and accuracy of the working level dilution. In addition, at least two independent standard sources must be used within an analytical sequence. While the laboratory control sample (LCS) and initial calibration verification (ICV) may be from the same source, a different source must be used for the daily instrument calibration standards. The continuing calibration standard (CCS) is usually prepared by serial dilution of the daily calibration stock standard source. If all CCV, ICV, and LCS criteria are met within an analytical sequence, the new standard lots are approved for use.

The 1000-ppm stock cyanide solution is prepared using KCN. The potassium concentration of the solution is verified by ICP analysis, which in turn verifies the cyanide concentration. This standardization is performed with each preparation of the stock solution. The primary standards used for standardization on the ICP are obtained from SPEX and are certified.

Preparations may be tracked through the date of preparation and chemist's initials, which are recorded on the standard bottle and in the standards logbook. Bottles are labeled and logbooks are completed as described above for GC/MS and GC standards. Copies of the preparation logbook page are provided with the raw data from the laboratory for verification by data reviewers and final technical reviewers.

All standards used by the Radiological Laboratory are purchased directly from NIST or from a commercial standards vendor, such as Amersham. All standards are NIST-traceable. A certificate is provided with each standard which contains information on the standard reference material number, the isotope, the activity, solution, and other key parameters.

The standard identification number is recorded in a logbook immediately upon receipt. Also recorded are the date received, the isotope, the certification number, the volume, and the original activity. All standards, both stock and working solutions, are stored in a single location, apart from samples.

The stock standard solutions are usually at high concentrations and are diluted to the working level solutions. The dilution factors are calculated based on the required activity levels and the activity of the original stock. All dilutions and calculations are recorded in a separate standards preparation logbook. Laboratory pure water used for dilutions is tested and verified to be radioactively inactive before use. Each working level standard is tested before use and the activity is recorded in the preparation logbook.

The working solution container is labeled with the name of the isotope, the date of preparation, and the standard reference number. Isotopes with the shorter half lives are decay

corrected based on the date used and the reference date on the certificate. Solutions are prepared as needed.

9.0 Document Control of Standard Operating Procedures and Laboratory Logbooks

Standard operating procedures (SOPs) are developed and used to implement routine QC requirements for all monitoring, repetitive tests and measurements, and for inspection and maintenance of facilities, equipment, and services. CompuChem's procedures are documented and housed in separate SOP collections.Analytical SOPs document specific analytical tests while nonanalytical SOPs document routine laboratory maintenance activities. Analytical SOPs are either sample preparation procedures (SPPs) or instrument procedures (IPs).

A table (Table 5-1) of the analytical methods upon which all SPPs and IPs are based appears in Section 5.0. Table 5-1 provides a good understanding of both the scope of our analytical procedures and how thorough the documentation and control of these procedures is at CompuChem. The documentation of procedures is considered to be critical to the assurance of data quality.

SOPs are distributed by area; each laboratory or administrative area receives its own set of SOPs. There are three areas at CompuChem in which complete analytical and nonanalytical SOP collections reside: the Technical Communications department, the Technical Information Center (managed by Technical Communications), and the QA department. The SOP collections reflect a sample's progression through the laboratories, from receipt by the Sample Control department to mailing of the resultant data package by the Report Preparation department. These SOPs are useful reference documents when employees are being trained, or when a question arises about an analytical or nonanalytical task.

9.1 Creating and Revising SOPs/Logbooks/Notebooks

The Technical Communications department staff are responsible for formalizing SOP drafts produced by laboratory or administrative area managers or senior staff members. Formalization of SOPs includes editing drafts, assigning document control data to each SOP, reproducing and distributing SOPs, and revising SOPs as needed.

SOP Review, Formalization, and Distribution

All new or revised SOP drafts must be reviewed and approved (by signature) by the author, a qualified second party in the author's area, a QA department representative, and a Technical Communications department representative. The author and the qualified second party reviewer examine the SOP to ensure that it accurately reflects the procedure as it is performed in the laboratory or administrative area. It then goes to the QA department where it is reviewed for technical accuracy, for adherence to the published method upon which it is based, and for compliance with associated contracts or regulations.

Once the SOP draft has received QA approval, it goes to the Technical Communications department, where trained professional technical writers review it, thoroughly edit it, and assign document control data to it so that the department is able to track all revisions of an SOP and to chronologically place any revision of the SOP. Such stringent document control is necessary because of the frequency of changes in

contract modifications, regulatory agency requirements, method revisions, and new analytical product offerings. The Technical Communications department then distrib utes the SOP to the area(s) in which it is used and to each of the three complete SOP sets. Each SOP recipient is required to verify receipt of it by signing and dating a New/Revised SOP form that accompanies all SOPs distributed. These forms are archived in the Technical Communications department office.

Document Control of Laboratory Logbooks

More than 200 different types of laboratory logbooks are used at CompuChem. To ensure document uniformity and compliance with U.S. EPA, good laboratory practices, and certifying agency protocol, the Technical Communications department has developed specific document control procedures for these vital QA records:

- all laboratory logbooks
- Inorganics and Organic Characterization Laboratory sample preparation logs and analytical runlogs

A laboratory logbook is developed cooperatively by the area manager and the Technical Communications department supervisor. The requestor submits a completed Logbook Request form to the Technical Communications department. They confer to design a prototype logbook page that both meets the needs of the laboratory and contains the key page elements required by the QA department. These elements are:

- the identity of the task
- the name CompuChem Environmental Corporation
- a "Reviewed By" signature field
- a date of review field
- any applicable measurement ranges with instructions for reporting out of range readings
- a corrective action statement
- model specifications for equipment

Next, the Technical Communications supervisor assigns an alphanumeric identifier to the logbook and produces one or more issues, each containing 150 pages of the logbook. The laboratory notifies Technical Communications when the logbook is ready to be archived. Before turning any logbook issue over to Technical Communications, the manager of the area in which the logbook is used or a designee must review the contents of the logbook. When approved, he/she signs the Logbook Authorization form, which is bound as the last page of each logbook issue.

When Technical Communications receives a logbook for archival, the supervisor of Technical Communications notes on the original Logbook Request form, the date of archival and the condition upon receipt for archival.

The issue is then logged into the Logbook Archival Log. After archiving the logbook, Technical Communications returns Inorganics Laboratory sample preparation logs and instrument runlogs to the laboratory for short-term storage (3 months), where they are used as reference documents. All other logbooks are placed directly into long-term storage in a secured, restricted-access facility off site. The supervisor of Technical Communications maintains a list of personnel authorized to enter the storage facility and to remove archived logbooks or runlogs.

Document control footers appear along the bottom of each page of each logbook, identifying the laboratory or administrative area, the logbook, and the issue of that logbook. In addition, each logbook is consecutively paginated and permanently bound. Each logbook contains a signature record for all personnel allowed to write entries in the logbook.

Logbook Retention and Record Purging

Logbooks are retained for five years from the date of archival. Logbook purging is recorded in the Archived Logbook Purge Log.

Laboratory Notebooks

Laboratory personnel do not have personal laboratory notebooks. Rather, each laboratory has a formal laboratory notebook which, once filled, is archived in the laboratory for easy reference. Technical Communications then replaces the filled notebook with a new one.

Laboratory notebooks are identified just as laboratory logbooks are identified and are prefixed with an "N" (e.g., N1A-1). Laboratory notebooks are checked in by Technical Communications as laboratories fill them. After notebooks are checked in, Technical Communications returns them to the area manager for storage and for use as a reference resource.

CompuChem's strict document control policies allow the Technical Communications department to account for all SOPs and logbooks, the period during which they were effective, and how and why each revision was made to each document. Equally strict control over document archiving and document custody ensures document accuracy and integrity. This document control program has been examined and approved by all state and certifying agency inspectors who have performed on-site system audits.

10.0 Data Reduction, Evaluation, and Reporting

10.1 Data Reduction for Organics Analyses

For analyses performed in the GC/MS Laboratory for volatile and semivolatile determinations, data is not read directly from the instrument but rather hardcopy output is generated through software programming. The hardcopy data is assessed through different tiers of review. Data files are transferred from the instrument through networking with the LIMS mainframe computer. Data files are verified to ensure consistency with the hardcopy. The hardcopy data generated from the instrument include the analyst worksheet, diagnostic report, quantitation report, compound list, chromatogram, internal standard response verification check, surrogate recovery form, target compound mass spectra-to-library comparison, tentatively identified compound (TIC) library searches, TIC worklist, and extracted ion current profiles. For analyses performed in the GC Laboratory for pesticide/PCB determinations, assessment is performed via terminal at the instrument.

All computer-generated compound lists containing the reportable results include formulas used to perform the calculations. These and other calculations are shown in Supplement D and are performed by instrument computers or qualified personnel. At least one extra significant figure is carried through all calculations until the final, reportable result is generated. Analytical results are never corrected for blank (background) contamination, but are flagged and footnoted appropriately.

Both the instrument operator and the data reviewer are responsible for determining that all calculations for surrogate recoveries and target compound concentrations performed by the instrument software are correct in that sample weights and volumes, final extract concentrations, dry weight factors, dilution factors, and amount of surrogate standard added were entered correctly into the formulas during software programming. Each GC/MS data system is capable of flagging all data files that have been edited manually. Any adjustments made to the hardcopy must be signed or initialled and dated by the reviewer. Data reviewers must assess all hardcopy data to properly interpret target compound mass spectra. TICs must be accurately characterized when compared to library searches and the assessment entered onto the TIC worklist.

Data reduction includes all processes that change either the instrument/computer-generated values, quantity of data values, or numbers of data items, and frequently includes computation of summary statistics. Documentation of the calculation process is required. In most cases, a programmable calculator, PC spreadsheets, or a computer program is used for calculations. The documentation allows the reviewer to verify the validity of the data reduction process.

All instrument and computer outputs contain a sample identification number (CompuChem number, or CCN) assigned by the LIMS upon sample receipt. This is a sequentially assigned, unique six-digit identifier which corresponds to the client field sample identifier. Data files contain both the CCN and the client-specified identification numbers.

All order entry information is entered into the LIMS by receiving clerks or customer account representatives. A backup of the hardcopy is stored in the Marketing department's project files. The information contains the project name, account number, and order entry number. Instrument operators program the instrument software to acquire data and generate hardcopy.

Data reviewers validate hardcopy results, making any changes or corrections. Data review clerks edit the LIMS data files to verify data file conformity to hardcopy results.

10.2 Data Reduction for Inorganics Analyses

In the Inorganics Laboratory, hardcopy output of the data is generated through computer software programming for all analyses except sulfate. For sulfates, absorbances are read and recorded directly from the spectrophotometer. These values are then manually entered into a computer software program that calculates the concentrations. The concentrations are then manually entered onto reporting forms. Data files are transferred directly from the instruments to the PDP-11 computer or stand-alone PCs using WARD software through networking for all ICP and GFAA metals, cyanide, mercury, and nitrate/nitrite analyses. Data files cannot be directly transferred for phenols and the wet chemistry methods performed on the Lachat. Phenol results are also manually entered into a computer software program that calculates concentrations. Results for phenols and wet chemistry methods performed on the Lachat are manually entered onto reporting forms. All manual data entries are verified by the data reviewers and final technical reviewers.

All final values are calculated by the computer software, including the linear regression calculations done to establish the calibration curves. When method of standard addition (MSA) is required for any of the GFAA metals, the linear regression calculations are performed by the analyst using a programmable calculator.

The laboratory technicians/chemists that prepare the samples are responsible for entering initial information into the computer such as the client identifier, sample weight/volume, pH, sample spike source, LCS source, and sample description, and for recording this information in their preparation logbooks. This information is used by the computer in combination with instrument results to calculate reportable values, and is verified by the data reviewer after analysis of the samples.

Preparation logbooks and instrument logbooks are used to verify final reportable values. This verification is performed by the data reviewer and again by the final technical reviewer. The logbooks are document controlled and kept in storage for future reference. Since all results are calculated using software programs, spreadsheets or worksheets are not necessary. All samples are identified by the CompuChem number, which corresponds to the client-assigned sample identifier. Computer data files are identified through this CompuChem number. Strip-chart recordings and hardcopy data outputs can also be identified by CompuChem number.

10.3 Data Reduction for Organic Characterization Analyses

In the Organic Characterization Laboratory, total organic carbon (TOC) final results are generated by the instrument software and are provided as hardcopy to the analyst. Values for total organic halides (TOX), total petroleum hydrocarbons (TPH), and oil and grease (O&G) are read directly from the instrument by the analyst. The analyst, in turn, enters specific information into the laboratory personal computer to generate final results using spreadsheets.

Information required for the calculation of final results from raw data includes: sample volume, absorbance, dry weight, dilution factor, and final volume. The analyst is also responsible for generating the final report that is sent to the client and for performing the first level of data review. The laboratory manager provides the second level of data review.

Preparation worksheets used in data reduction are bound and subjected to document control monthly. Bound worksheets are kept in secured storage for future reference. Copies of the worksheet page are included with each data package. All samples are identified by the CompuChem number. Each set of samples received from a client is assigned a unique case number. Hardcopy data (including strip chart recordings) for the TOC analysis is identified by CompuChem number and is filed with the associated worksheet.

10.4 Data Reduction for Radiological Analyses

Instruments used for radiological analyses output are used in data reduction. The Omnigam produces a computer-generated report for gamma analysis. A Region of Interest report for alpha and gamma analysis reports net and gross counts around a specific energy level. The Tennelec report generation includes a planchet number, counts, count time, and date. The Ludlum cell counter output consists of calculator tape.

Spreadsheets are used for data reduction by the radiological chemist performing the initial level of data review. Reduction from gross counts to picocuries requires the consideration of many variables. Each spreadsheet is specific to the analysis and uses appropriate formulas, times, efficiencies, decay factors, and conversion factors to perform the reduction. Results are transferred to the Radiometric Analysis Results form by the data reviewer. The final technical reviewer verifies that results are correctly reduced and reported.

10.5 Data Processing

This section summarizes the manner in which all aspects of data processing are managed and evaluated to maintain data integrity and characterize data quality. These processes include data collection, verification, transfer, and storage. CompuChem is committed to maintaining client confidentiality throughout the course of data generation.

Collection

Analytical data are generated from the GC/MS computer software, GC computer, ICP computer, atomic absorption spectrophotometers, autoanalyzers, and other laboratory instrumentation. The outputs include identifications of analytes, concentrations, retention times, and comparisons to standards. Outputs are in graphic form (chromatograms), bar graphs (spectra), and printed tables. The outputs are in standard format specified for each analysis type and are monitored for consistency. If incomplete or incorrect output is generated, corrective actions are taken according to SOPs established for each type of analysis. Corrective actions are consistent with the manufacturer's recommendations.

Most outputs are generated through computer programs that have been validated by Systems and Laboratory Automation department support before being used. The instruments have programs available for the analysts to manually verify integrations and quantitations. Manual verification is performed when there are near-eluting constituents or irregular baselines. In the review process, the data are compared to information about the sample processing history, sample preparations, sample analysis, associated QC data, etc., to evaluate the validity of the results.

Ancillary data that are produced for internal records and which may not be required by the customers as part of the analytical data package include the following:

- laboratory worksheets
- associated quality control sample data
- sample tracking system forms
- standard preparation records
- instrument logs
- calibration records
- maintenance records
- laboratory logbooks

These data are available for inspection during audits to verify the validity of data and are also deliverable, depending on the client's needs. A complete record of each sample's history is available for documenting its progress through the laboratory from sample receipt to reporting. Document control and COC requirements include additional information about documentation and archiving of data.

Review and Verification

Data verification takes place on two levels. First, the QA department is responsible for monitoring all laboratory QC activities and for verifying that systems are in control. QA's responsibilities and the manner in which QA fulfills them are described in the QA SOPs as well as this QA Plan. The QA department therefore plays a role in data verification in the context of the overall QA program.

Data verification also occurs on a sample-by-sample basis. This occurs during the various levels of data review that take place within the laboratory. The first level of review occurs at the bench. This initial review by the instrument operator or analyst includes:

- cross-checking all sample identification numbers on worksheets, sample preparation logs, extract vials/digestate bottles, and instrument outputs
- calculating surrogate recoveries and internal standard responses (when applicable) and verifying that QC acceptance criteria are met
- verifying that all calibration, tuning, linearity, and retention time drift checks are within QC acceptance criteria
- verifying that all target analytes are within the instrument's analytical range and determining appropriate dilutions when necessary

- determining that peak chromatography and other instrument performance characteristics are acceptable

- verifying that COC is intact based on accompanying paperwork

The second level of review is performed by the in-lab data review staff. In the GC/MS Laboratory, these reviewers are experienced mass spectroscopists trained and qualified to interpret mass spectra. GC Laboratory and Inorganics Laboratory data reviewers have college degrees and are senior level chemists. Senior data reviewers, the manager of data review, or a laboratory manager also audit a percentage of these data before they are released to the Report Preparation department. In-lab data reviewers verify all assessments previously made by the operator/analyst, and also:

- verify that all quality control blanks meet QC requirements for contamination, and that associated sample data are appropriately qualified when necessary

- calculate matrix spike recoveries and duplicate RPDs, and verify that accuracy and precision QC criteria are met

- compare all injections of a sample and compare matrix spikes with the original unspiked sample for acceptable replication

- qualitatively identify all target analytes using specific SOP interpretation criteria

- verify computer quantitation of all target analytes, and evaluate Extracted Ion Current Profiles (EICPs) and chromatograms for proper resolution and integration, when necessary

- verify that analytical worksheets and preparation and instrument logs, have been correctly completed by the operator/analyst, including date and initials

- verify for pesticide GC/MS or GC confirmation analyses that target analytes were within retention time windows and/or evaluate spectra for proper identification, and compare to initial analysis

- for GC/MS analyses, evaluate Library Search mass spectra, characterization of TICs, and verification of calculations for estimated concentrations of these compounds

- verify that good laboratory practices were followed relative to the correct procedure in making changes to data

The completed data package, which has been reviewed on an analytical fraction basis (i.e., volatiles, acids, base/neutrals, pesticides), is then forwarded to the Report

Preparation department. The package is integrated with other fractions from the same sample, and with associated deliverable items as required by the client. The assembled package is forwarded to the Final Technical Review department staff for the third level of review. The final technical reviewer, also a senior chemist and experienced data evaluation specialist, assesses the complete data report (called a case or SDG, for CLP format reports) and double-checks all items previously verified by the in-lab data reviewer. Additional assessments include:

- reviewing all data summary documents and verifying correct transcription from raw data
- making a comparative evaluation of data from individual fractions of a sample, and of samples from the same site, project, or case for consistency of analytical results and resolution of discrepancies
- checking the data report or case for completeness, including requirements for the complete sample delivery group (CSF)
- For CLP-format reports, writing a case narrative that authorizes release of the data, provides end-users with a history of the sample processing, documents the quality control process used and exceptions to SOW criteria, and summarizes any corrective actions taken

Data Transfer

Data transcriptions for final reports to clients are performed by Report Preparation department clerks. For non-CLP reports, the reportable data is reviewed and approved by the final technical reviewer, then word processed by computer. Verification of the wordprocessing function is performed by a proofreader before the data is released. For CLP reports (whether to EPA or commercial clients), all raw data are reduced into deliverable format by Report Preparation department clerks, who also summarize data onto forms required by the CLP SOW. The clerks use a PC-based software system that extracts data directly from the laboratories' computers. The final technical reviewer is provided with both the deliverable report and the non-deliverable back-up data, and must verify the accuracy of all transcription processes.

When all levels of review have been completed and data release has been authorized by the final technical reviewer, the data report (or case of reports) is sent to the copy center for mailing. For EPA, the complete sample delivery group (CSF) is assembled and must contain:

1. inventory sheet
2. SDG case narrative
3. Traffic Reports
4. volatiles data
5. semivolatiles data
6. pesticides data
7. miscellaneous data
8. EPA shipping/receiving documents
9. internal lab sample transfer documents
10. other records

All data in the CSF must be paginated. This is done in the copy room by the deliverables clerks. Items 2-6 are part of the sample data package and are paginated in ascending order. Items 7-10 are paginated in ascending order beginning with page number 10000. The page numbers are entered on the DC-2 Form, which is used to verify consistency and completeness of the case.

Copies are sent to EPA's Environmental Monitoring Systems Laboratory (EMSL-LV) and to the Sample Management Office. COC seals, signed by the deliverables clerk, are used to secure all deliverables packages. Items 1-10 (above) are sent to the regional client .

Senior members of the QA department are also required to audit approximately 5-10% of all analytical data. The QA auditor performs the same assessments as the final technical reviewer. Findings from these data audits are presented in a report to management.

Data Storage

At every stage of data processing during which a permanent collection of data is stored, procedures are established to ensure data integrity and security. Specific QA Project Plans indicate how specific types of data are stored with respect to media, conditions, locations, retention time, and access. Table 10-1 presents general guidelines. (Clients may request that we retain magnetic tape for an extended period.)*

Table 10-1. Guidelines for Data Storage

Media	Conditions	Location	Retention Time	Access
Hardcopy**	Locked warehouse	off-site	5 years	Document Custodian or other designated personnel
Magnetic Tape	Locked storage (controlled environment)	on-site	5 years 1 year (EPA)	Document Cust. or other designated personnel

*After five years, hardcopy data will be shredded and recycled or returned to the client. Return shipment is at the client's expense and will be done only if the client notifies us when the project is set up and the first COCs arrive. The CompuChem COC form contains a field to indicate data disposition after the retention period. This allows us to specially mark and isolate storage boxes and folders.

**Unless otherwise directed by the client.

Hardcopy data is indexed for retrieval by Case Number / Project Number. These numbers originate from client account/order numbers for non-EPA clients. Hardcopy data includes all data generated for the required analysis, including ancillary data, such as extraction logs, which may not be required as part of the reportable analytical data package, Procedures used to maintain electronic data are discussed fully in Section 7.0 of this QA Plan.

The document control clerk, who reports to the document control officer, is responsible for maintaining custody of and an inventory of completed EPA and commercial folders. From this inventory, the control clerk is often asked to pull cases and folders for data inquiries. In addition, the clerk stores the documentation of completed sample analyses in the local warehouses used by CompuChem Environmental Corporation. To be able to produce reports with sample results and data on request is important to CompuChem and to our clients.

11.0 Quality Control Samples and Documentation

The analytical and QC requirements for each sample are met with the help of CompuChem's LIMS. The LIMS is operated by one of the mainframe computers and is accessible from any of more than 90 CRT terminals. The LIMS is based on analysis codes defined to schedule appropriate analytical procedures and QC samples required for each batch of samples.

Associated with analysis codes are LIMS-defined instrument procedure (IP) codes, sample preparation procedure (SPP) codes, and QC counters that allow the LIMS to track samples and analyses and to control the frequencies of QC samples. The IP and SPP codes are directly linked to the laboratory SOPs and analytical worksheet for each procedure. QC counters define the types and frequencies of QC samples associated with each batch, and are determined by method, contract, QAPjP, and/or the associated U.S. EPA CLP SOW. When specific methods are required, such as CLP organic and inorganic SOW methods, EPA's *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*, SW-846 third edition, or *Federal Register* 40 CFR 136 methods, QC counters are defined to produce the types of QCs at the frequencies specified in Tables 15-1 through 15-4. For frequencies of QC checks, refer to Section 8.0.

Additional QC check standards may be included and shall be used if specified by the approved method:

- reagent purity checks
- internal standards
- surrogate spikes

Trip blanks: For VOA analyses, at least one trip blank preserved to pH < 2.0 is prepared and analyzed for each cooler used for storage and transport of samples. It is the responsibility of the client to order trip blanks as needed.

As an additional measure, the QC counters are defined to provide for the additional preparation and analysis of an LCS or blank spike, which is used to evaluate laboratory performance and sample matrix interferences for each batch. Counters may be redefined to allow for specific requirements of a QAPjP, state certifying agency, or U.S. EPA region. Only the VPGM has final authority for altering or creating new QC counter definitions.

11.1 Surrogate and Spike Standard Recoveries

The precision and accuracy of a method as applied to a specific sample matrix may be assessed by evaluating the recoveries of spike and surrogate standard analytes. The recovery of the spiked analyte is used to assess the method accuracy. When sample duplicates or MSDs are performed, the relative percent difference (RPD) between the recoveries of the spiked analytes may be established for use in assessing method precision. A standard solution containing a minimum of three system monitoring compounds is added to each sample requiring GC/MS analysis for volatile organic compounds. A minimum of three surrogate standards, but generally six, are added to each sample requiring GC/MS analysis for semivolatile organic compounds.

Pesticide analyses require at least two surrogates and herbicide analyses require at least one. Inorganic and organic matrix spikes and blank spikes (LCS) are similarly fortified with spike standard solutions containing target analytes of interest. The recovery of these standards is quantitatively measured during analysis, and historical records of the percent recovery (%R) for each sample are maintained in a database. Surrogate and spike compound recoveries must meet acceptance criteria before the analytical data will be released. In some instances the sample matrix may produce interferences that adversely affect recoveries. Surrogate recovery interferences must be confirmed by repreparation and reanalysis of the sample. When a matrix spike test fails spike recovery criteria, the LCS must be evaluated to determine whether the spike failure is sample matrix related. If the LCS test fails, the associated sample data must be inspected or the batch reprocessed. If the LCS passes, the matrix effect is confirmed and the affected data are qualified by a QA Notice.

Depending on the type of problem identified and on the sample holding time, corrective action may involve reporting data as is with a laboratory qualifying notice. An example is the poor precision between matrix spike duplicates when one of the extracts is not concentrated accurately. When an analyte does not meet criteria for RPD in inorganic duplicates, a data qualifying flag accompanies the analyte result in associated samples in accordance with the U.S. EPA CLP reporting convention. Samples are fortified before extraction, purging, and digestion or distillation. Laboratory duplicates are prepared from field samples for inorganics analysis, and RPDs are calculated. The recoveries of surrogate and spike standards in the LCSs are also quantitatively measured. LCS recoveries are maintained in a control chart database. The statistical warning and control limits are updated periodically.

11.2 Laboratory Control Sample / Control Charting Program

Surrogate and spike recoveries of LCSs are monitored using control charts. Corrective action is taken at the instrument for out-of-control data points and is documented on a control chart evaluation form. An LCS is prepared and analyzed with every batch of samples for each matrix and method performed by the laboratory. All LCS data are collected daily, and surrogate or spike compound recoveries are entered into a database used for printing control charts for each monitored compound. Evaluations are made by laboratory managers and chemists who determine what events necessitate that real-time corrective actions be taken. The LCS is always prepared from a different stock standard source than that used for the analytical calibration standards.

The LCS used in aqueous volatile GC/MS analyses is the instrument blank, analyzed every 12 hours at the beginning of a calibration period. Laboratory pure nitrogen-sparged water is fortified with surrogate (system monitoring) compounds. Recoveries are plotted on control charts. The LCS used in solid GC/MS volatile organics is the method blank prepared by spiking commercially prepared laboratory blank sand with surrogate (system monitoring) compounds. The method blank follows the samples through the entire preparation and instrument analysis procedure.

The LCS used for aqueous and solid GC/MS semivolatile analyses is the method blank, which is prepared by spiking the appropriate laboratory pure matrix before extraction, and which follows samples through the analytical process. Surrogate recoveries are plotted on

control charts.

The LCS for pesticide/PCB methods by GC analysis is prepared by spiking the appropriate laboratory pure matrix with surrogates and selected target analytes. Surrogate and spike analyte recoveries are plotted on control charts. Non-pesticide GC methods (herbicides, volatile organics, and PAHs) use only surrogate recoveries for control charting purposes.

Aqueous inorganic analyses require the use of laboratory pure water fortified with a certified reference standard. The LCS is subsequently digested or distilled and analyzed with each batch of samples. It contains all target analyte elements required of the U.S. EPA CLP. The solid LCS consists of a certified, commercially supplied homogenized material fortified with the full target analyte list of elements.

Organic characterization methods for TOC/TOX include an LCS that is a fortified laboratory pure matrix. The extraction fluid, Freon-113, is used to fortify the LCS for TPH and oil and grease determinations . Each organic characterization LCS contains those components found in the calibration standards. Radiological methods use a fortified pure blank matrix and are plotted for each radioisotopic analysis performed in the Radiological Laboratory.

Information provided to the database is printed onto summary forms and includes preparation date, extractor ID number, date of analysis, instrument ID number and compound number ID. This information assists the laboratory in determining trends or systematic error.

The laboratory uses EPA method-required limits or statistically generated limits based on actual laboratory performance data for every LCS analysis. For CLP methods, limits are updated to statistically derived limits only if the laboratory control range is tighter. As control limits are updated for each method based on actual laboratory performance data, the control limits and warning limits are statistically determined at three and two standard deviations, respectively.

Both I-Charts and R-Charts are provided. The I-Chart plots individual surrogate or spiked analyte recoveries, while the R-Chart plots the range (difference) between recoveries of successive blank spikes. The Western Electric Pattern Rules are used by the Quality Analyst software program* to test each datapoint for rule violations. The software flags points in violation of these rules or any data point falling outside the 2-sigma warning limits or 3-sigma control limits.

Certain pattern rules indicate warning conditions that alert the laboratory personnel to a potential out-of-control condition. The laboratory must evaluate the next acquired data point and determine whether corrective action is required. Other pattern rules indicate that a potential trend or systematic error could be occurring.

11.3 Quality Control Checks for Sources of Contamination

A method blank is prepared at the frequency specified by the method. The purpose of the method blank is to ensure that contaminants are not introduced by the glassware, reagents, standards, personnel, or sample preparation environment. For volatile analyses, an instrument

*Quality Analyst is a registered trademark of Northwest Analytical, Inc.

blank is also analyzed during each calibration shift to verify that contaminants are not being introduced by components of the instrumentation or analytical laboratory. Criteria for the evaluation of these blanks are presented in Section 15.0.

The following routine QC checks are performed to verify that samples are not contaminated during transportation, preparation, analysis, or storage, and that standards prepared internally are traceable to certified sources:

- water purification systems check
- refrigerated storage system checks
- reagent and solvent purity checks
- standards preparation and traceability checks

The criteria for these QC checks and corrective action steps are detailed in QA SOPs. Results of these checks are audited by the QA department and corrective action is taken as needed.

The quality of the laboratory water and the cleanliness of the glassware used to prepare samples is of utmost importance in preventing contamination. Therefore, a brief description of the laboratory water system and the glassware cleaning procedures follows.

11.4 Laboratory Pure Water System

Instruments are now capable of detecting and measuring trace elements down to the level of fractional parts per billion. Results would be meaningless if background contaminants in the laboratory pure water masked the very elements being analyzed. To eliminate this problem, CompuChem produces ASTM Type I reagent grade water (which is used to prepare method blanks, blank spikes, instrument blanks, reagents, and standards) with an Ionpure reverse osmosis pure water system (model Milli RO 350). The system has a storage capacity of 275 gallons. A stainless steel pump continuously recirculates the water at 12 gallons per minute through two mixed-bed deionizers, an ultraviolet sterilizer, and the pure water loop. Water is fed to the glassware preparation area, the Inorganics Sample Preparation Laboratory, the GC Laboratory, the Radiological Laboratory, the TCLP Laboratory, and the GC/MS Volatiles Laboratory through 800 feet of 1.5-inch polypropylene pipe. The water is recirculated back to the storage tank through the mixed-bed deionizers, the ultraviolet sterilizer, and the pure water loop.

The Ionpure system runs automatically and consists of:

- an automatic backwashable carbon tank that removes organics, chlorine, and sediments
- an automatic regenerable water softener tank
- polycord five- and one-micron prefilters to remove particles from the city tap water
- Milli RO 350 six-bowl system

- a 275-gallon fiberglass tank which includes a 0.22 μm hydrophobic cartridge to remove particles and microorganisms from gases and liquids
- a stainless steel centrifugal pump rated for 12 gpm
- two mixed-bed deionizers rated for 15 gpm to remove dissolved salts and minerals that generate water up to a maximum of 18 megaohm/cm resistivity at 25°C
- an in-line indicator/controller that reads solution resistivity at 25°C
- an ultraviolet sterilizer with two UV lamps and 20-gpm capacity

The Milli RO 350 requires a minimum of 40 pounds per square inch of water pressure to operate. The system starts and stops automatically, based on water level in the 275-gallon storage tank. The system is monitored daily and readings are recorded in a logbook. When the resistivity reading of the first mixed-bed deionizer falls below 10, the tank is exchanged. To produce organic-free water for HPLC analyses, a Compact Milli-Q Plus polishing system is added in the GC Laboratory. This system contains organics-scavenging cartridges that reduce the total organic carbon (TOC) levels to less than 50 ppb without ruining resistivity. A Barnstead polishing system is used in the Inorganics Laboratory to ensure the water measures at least 16.8 megaohm/cm resistivity. The water is sparged with nitrogen for 24 hours before use in volatile organics analysis by GC/MS or GC.

11.5 Glassware Cleaning Protocols

Preparing Glassware for the Organic Sample Preparation Laboratory (Organics, Acid B/N Extractables, and Pesticide/PCBs)

All sample processing glassware is cleaned thoroughly as soon as possible after use. Before being washed, dirty glassware is rinsed by the extractor with the last solvent used. The glassware is then washed with hot, soapy water, using a phosphate-free, biodegradable detergent such as Contrad-70. All glassware is then thoroughly brushed, and all brushes used are subsequently rinsed.

Glassware is then rinsed with tap water, then rinsed once and sprayed once with deionized water, and then drained. Glassware (with some exceptions) is then annealed at 500°C for four hours. When glassware is removed from the oven, it is checked for stains and breakage. If either staining or breakage has occurred, the glassware is discarded or, when possible, sent for repair. Glassware is stored on trays in the glassware preparation area.

Preparing Glassware for the Inorganics Laboratory

All sample processing glassware is cleaned as soon as possible after use and then washed in a series of water and/or acid treatments depending on the type of glassware.

Beakers: Each beaker is rinsed thoroughly with DI water, thoroughly brushed, and rinsed again with DI water. The beakers are then soaked in 50% HNO_3 solution in a Nalgene tub for at least 30 minutes. Beakers are removed and drained, then soaked for five minutes in a sink of DI water. Beakers are removed and rinsed twice with DI water. A 100-ml portion of 5%

HNO_3 solution is then poured through each beaker sequentially. The solution is taken from the last beaker and tested on ICP and GFAA. Each analyte must be below the CRDL or the beakers must be cleaned again and tested again. Clean beakers are stored covered on carts.

Graduated cylinders and fleakers: Each cylinder is filled with DI water and scrubbed with a brush, then placed in a 50% HNO_3 bath for at least 10 minutes. Then they are drained and soaked in DI water for at least five minutes. Each cylinder is then rinsed with DI water and stored in a cabinet.

Volumetric flasks: Approximately 30 ml of 50% HNO_3 solution is added to each flask. The solution is swirled in the flask and then dumped into the acid waste container. Each flask is then rinsed with DI water four times and drained. The flasks are stored in a glassware cabinet.

Cleaning the Zero Headspace Extractor (ZHE) and Associated Glassware (Beakers, Flasks, Graduated Cylinders, Syringes)

The glassware used in preparing extraction fluid and 1.0 N sodium hydroxide, the syringes used to collect the ZHE extract, and the beakers and graduated cylinders used for ZHE preparation are washed with hot soapy water using a phosphate-free biodegradable detergent such as Contrad-70. The glassware is then rinsed with tap water and with DI water, then heated at 500°F for one hour. The glassware is then moved to the Volatile Sample Preparation Laboratory to cool.

The ZHE apparatus is taken apart in the glassware preparation area and the waste is emptied into hazardous waste containers. Each component of the ZHE is washed with hot, soapy water using a phosphate-free biodegradable detergent such as Contrad-70, then rinsed with tap water, and, subsequently, with DI water. The ZHE screens are then heated at 500°C for one hour, and then moved to the Volatile Sample Preparation Laboratory to cool.

After the screens have cooled and the pistons and the bottom portion have been inserted, two rinsing steps are performed. First, 200 ml of methanol are poured into the cylinder and the top of the ZHE is assembled without the filter, but with the screen, and pressurized. The valve is opened so that the methanol can drain. This rinse is performed twice. Then, 500 ml of sparged DI water is added. The ZHE is pressurized, and the valve is opened to release the water. This rinse is repeated and the ZHE apparatus is ready to be used.

Preparing Glassware for GC and GC/MS Volatile Sample Preparation

All glassware is cleaned thoroughly and as soon as possible after use. Glassware is washed with hot, soapy water, using a phosphate-free biodegradable detergent such as Contrad-70. Glassware is then rinsed with tap water and with deionized water, and drained. Glassware (except volumetric glassware) is heated at 500°F for at least one hour in a conventional oven. Glassware is then allowed to cool in a contaminant-free environment before use.

Preparing Glassware for the Radiochemistry Laboratory

All glassware used in the Radiochemistry Laboratory is cleaned as soon as possible after use. Glassware is washed in Radiacwash detergent and water. Rad-Con is used on tough stains and on glassware that is known to be contaminated. Glassware is thoroughly brushed, and the

brushes are subsequently rinsed. The glassware is then rinsed with tap water. Any glassware that was baked during use (any procedure in which the glassware's contents were evaporated to dryness) is placed in an 8N HNO_3 acid bath for one hour. This acid bath is changed at least biweekly. Following the acid bath, glassware is rinsed with tap water, then rinsed twice with DI water before being drained.

11.6 QC Standards Preparation Checks

All calibration standards are traceable to the National Institute of Standards and Technology(NIST) or EPA-certified standards whenever such standards are available. Commercial sources of standards and reagents are checked for purity and are approved before being used in sample preparation and/or analysis. All standards used in the analysis of samples under the current CLP Statement-of-Work (SOW) are purchased with certificates of purity and traceability.

All organic standards prepared for use throughout the laboratory are assigned two code numbers, one identifying the standard type and a second identifying the individual preparation lot. The standard code numbers are entered in a bound standard preparation logbook with all information regarding the preparation of that standard (i.e., date, technician, name of each compound and amount used, final volume, solvent used, and a vendor code). The vendor code traces the preparation of that lot to the vendor supplying the standard material, the vendor's lot number, and the particular bottle or ampule from which the material was taken. All containers for standards are labeled with the identification code and lot number code, initials of the technician, and the date of expiration.

The instrument response obtained for each compound in a newly prepared standard is compared to the response obtained from the previously approved standard. The two standards' relative response factor (RRF) ratios (test rrf/reference rrf) must agree within ±15% warning limits or ±20% action limits (for all but a few compounds recognized as being chromatographically atypical), or the new standard may not be used until the discrepancy has been resolved.

The working lifetime of standard preparations are dependent on the compound types comprising the standards. Shelf life of standards is determined during storage stability studies carried out by the Standards Laboratory. Most standards are prepared with far greater frequency than recommended by the EPA, and in no case is the recommended frequency exceeded.

11.7 External Reference Standards

Continuing calibration and calibration verification standards ("calibration checks") are used during each calibration period to demonstrate that the instrument's standard curve still meets QC criteria. These standards are usually prepared from a different source than those used for the instrument's initial calibration curve. A matrix spike and/or blank spike is used to further demonstrate that the entire analytical system is in control.

QC reference standards, used in the single blind performance evaluation program, are analyzed at least once per quarter for additional verification with external standard sources. All continuing calibration and calibration verification standards and external reference standards are obtained from EPA or are traceable to NIST- or EPA-certified standards (when available).

11.8 Specific Routine QC Requirements

Because of the large number of parameters and potential sample matrices, it is difficult (and impractical, under most applications) to develop precision and accuracy objectives and control limits for every parameter in every matrix. Therefore, it is necessary to extrapolate this information from a limited number of parameters and/or matrices. The laboratory accomplishes this by using surrogate standards in all organic sample analyses, by spiking randomly chosen samples with various target analytes, and by producing an LCS (spiked blank matrix) with each batch of samples. An initial one-time demonstration of precision and accuracy is made using replicate blank spikes as part of a method validation.

The QA department plots control charts for LCSs (blank spikes) and blanks daily. For all CLP analyses, precision and accuracy data are required to be tabulated and reported on the "MS/MSD Form III." These data are then statistically analyzed by the U.S. EPA (Environmental Monitoring Systems Laboratory-Las Vegas), and presented periodically to all CLP laboratories in Laboratory Profile Packages. In this way, both intra- and inter-laboratory trends in QC results can be observed.

The following sections describe the primary QC requirements for both organic and inorganic analytical programs.

Organic Program QC Requirements

GC/MS Calibration: The GC/MS instruments must first be standardized (for mass assignment) according to the manufacturer's procedures using a standard called FC-43 (perfluorotributylamine). Once every 12 hours the GC/MS instrument is "hardware tuned" using either decafluorotriphenylphosphine (DFTPP) or bromofluorobenzene (BFB), depending on the type of analysis being performed. This procedure assures that other instruments both within and outside the laboratory will be operating under similar conditions, and assures comparability of mass spectral data generated under those conditions.

The mass spectrum from the DFTPP or BFB analysis must meet the method-specified criteria such as those described in the U.S. EPA CLP Statement-of-Work. These criteria are comparable to those specified in EPA's *Test Methods for Evaluating Solid Waste: Physical/ Chemical Methods*, SW-846 third edition, and those in 40 CFR Part 136. For the analysis of the semivolatile extractable compounds, DFTPP is used in tuning the instrument. For the analysis of volatile organic compounds, BFB is used in tuning the instrument.

Specific ion abundance criteria for the tuning compounds are listed in Section 8, Tables 8-1 through 8-4. The bar graph mass spectrum and mass listing serve to document the proper tuning of the GC/MS system. Once the instrument has met key ion abundance criteria for the tuning compounds, the GC/MS is calibrated. Calibration curves are generated as outlined in the EPA CLP SOW and in the *Federal Register* 600 series methods. After the initial calibration curve is established using several different standard concentrations (as specified in the

method), the calibration linearity and system performance are verified every 12 hours by analyzing the tuning compounds and continuing calibration standard. If significant variation in compound RRFs or loss of instrument sensitivity has occurred, a new initial calibration curve must be generated. The criteria for determining acceptable continuing calibration responses are outlined in the EPA CLP SOW, the *Federal Register*, and EPA's *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*, SW-846 third edition.

The analyst may not proceed with sample analyses until the instrument has met tuning requirements and the continuing calibration standard is shown to be within specific criteria when compared with the initial calibration curve (see Section 8.1). *GC Calibration*: The GC instrument (with EC detector) is calibrated for pesticides/PCBs analysis as described in the EPA CLP SOW, and in EPA's *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*, SW-846 third edition, and 40 CFR Part 136. Each time a new initial calibration is required and each time a new GC column is installed, a determinationof retention time windows for each target analyte and surrogate is made. The calculation used for retention time windows is method-specific. These retention time windows are used to make tentative identification (followed by a confirmation analysis on a dissimilar column). These data are retained by the GC Laboratory and made available during on-site laboratory evaluations.

The external standard method is used for all analyte and surrogate quantitations. Once the initial retention time windows are established, the laboratory may proceed with the routine calibrations following the EPA CLP SOW, EPA's *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*, SW-846 third edition, and 40 CFR Part 136.

Certain evaluation standards and individual standards are used to verify instrument linearity, Endrin and/or 4,4'-DDT degradation, retention time shift and windows for that sequence, and instrument stability (based on variations in calculated calibration factors for each target analyte) over the course of the sequence. Details of the composition and calibration of these standards are presented in the EPA CLP SOW, in the GC Laboratory SOPs, and in Section 8.1 (Tables 8-5 through 8-6) of this QA Plan.

Method Blank/Instrument Blank Assessment: CompuChem's policies for allowable levels of contamination are more stringent than those specified in the CLP SOW. For common laboratory solvents (methyene chloride, acetone, and phthalate esters) the maximum allowable level of contamination in method or instrument blanks is twice the contract-required quantitation limit (rather than the 5X CRQL allowed in the CLP SOW), with certain exceptions

For the remaining volatile, semivolatile, and pesticide target analytes, the concentration in a method or instrument blank may not exceed one-half the CRQL (see Section 15.0 for more detailed information). These internal criteria are waived if holding times are in jeopardy of being exceeded.

Precision and Accuracy Objectives for Organic Analyses: Organic surrogate recoveries are used to determine whether the sample processing and analysis functions are in control. With few exceptions, surrogate recoveries must be within control limits or the sample processing and analysis must be repeated. One exception involves the pesticide surrogates tetrachloro-m-xylene and decachlorobiphenyl, which are used for advisory purposes only (as directed in the EPA CLP SOW), although recoveries must be greater than 20%. The other exception involves the surrogates for acid and base/neutral extractables: for CLP analyses, no more than one surrogate from each fraction (acid or base/neutral) may be outside control limits.

Matrix spike control limits for organics samples associated with the U.S. EPA CLP are also for advisory purposes. Samples processed following procedures designated in 40 CFR Part 136 and those associated with EPA's *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*, SW-846 third edition, must meet acceptance criteria specified therein. The EPA CLP methods require the calculation and documentation of relative percent difference (RPD) between recoveries of the matrix spike (MS) and matrix spike duplicates (MSD), although acceptance criteria are also advisory. CompuChem has adopted internal accuracy and precision criteria to be used as decision guidelines where the contract provides advisory criteria as follows.

For MS/MSD tests based on methods from EPA's *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*, SW-846 third edition, and from U.S. EPA CLP SOWs, at least one half of the QC spiking compounds must be recovered within acceptance criteria for each organic fraction. Similarly, at least one half of the precision criteria (RPDs) must be met per analytical fraction. If these criteria are not met, the matrix spike and matrix spike duplicate tests have to be repeated unless a sample matrix effect is confirmed with the original unspiked sample. For *Federal Register* requirements, a matrix spike, fortified with the full complement of target analytes, is performed for organic analyses. A blank spike (LCS) is also processed with the batch. If all compounds in the matrix spike are not recovered within acceptance criteria, the blank spike is analyzed . If neither QC sample meets criteria, the entire batch is reprocessed, unless limits are advisory and the holding time has expired. Precision and accuracy acceptance limits for CLP organic and inorganic analyses are contract-mandated. Depending on the CLP SOW, those same criteria have been incorporated into EPA's *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*, SW-846 third edition.CompuChem also offers a variety of analytical services using *Federal Register* methods. The QC requirements for accuracy and precision are also method-mandated. In 40 CFR Part 136, it is recommended that the laboratory periodically update these control limits based on historical data. Updated control limits will be based on the following formulae:

$$LCL = X - 3SD$$
$$UCL = X + 3SD, \text{ where}$$

LCL = lower control limit
UCL = upper control limit
X = mean percent recovery
SD = standard deviation

Precision and Accuracy Objectives for Inorganic Analyses: The inorganic program QC requirements are similar to those outlined above for the organic program. Metals, except mercury, are analyzed using flame and furnace atomic absorption (AA) spectroscopy and/or inductively coupled plasma (ICP) spectroscopy. The analysis procedure generally involves two steps: sample digestion and subsequent instrumental analysis.

The quality of these results is assured by several key procedures. Although surrogate standards are not applicable to inorganic analyses, the laboratory uses sample spikes and duplicates in much the same way as the organic program to assess data accuracy and precision. In addition, an LCS and a method blank are produced with each batch of samples. These QC samples are involved in both the sample digestion and analytical processes, and represent the conditions under which associated samples were processed.

Calibration standards are obtained from the U.S. EPA and other independent certified sources. Inorganic standards are prepared by the Inorganic Laboratory analysts, and the preparation is documented in the laboratory's standard preparation and traceability logbooks. The standard container is labeled with the preparer's initials, date of preparation, and type of standard.

Instruments are calibrated following the requirements set forth in the U.S. EPA CLP Inorganic SOW, EPA's *Test Methods for Evaluating Solid Waste: Physical/ Chemical Methods*, SW-846 third edition, or other EPA-approved methods, as described below and in Section 8.1 of this QA Plan. In-lab data reviewers and final technical reviewers use guidelines documented in their respective SOPs for verifying compliance of calibration data with QC requirements.

AA and ICP Instrument Calibrations: For inorganic analysis by AA and ICP spectroscopy, inital calibration is performed using dilutions of stock metal solutions. For AA calibration, a blank and at least three calibration standards are employed. ICP calibration is performed in accordance with the instrument manufacturer's recommendation. For ICP analysis this includes, at a minimum, a blank and a midconcentration level standard.

After the AA and ICP systems have been calibrated for every analyte, the initial calibration must be verified for accuracy. This is accomplished by immediately analyzing an EPA or EPA-approved initial calibration verification solution at a concentration other than that used for calibration, but within the calibration range. An independent standard is one prepared from a different source than those used in the initial calibration.

To assure calibration accuracy during the course of sample analysis, a continuing calibration verification (CCV) standard is analyzed at a frequency of 10%, or every two hours during the analysis run, for each analyte. The analyte concentrations in the continuing calibration certificaton standard are near the midrange level of the calibration curve. The initial and continuing calibration verification control limits are listed in Table 11-1. A continuing calibration blank (CCB) is analyzed after each CCV.

Table 11-1. Initial and Continuing Calibration Verification Control Limits for Inorganic Analyses

Analytical Method	Inorganic Species	% of True Value (EPA Set) Lower Limit	Upper Limit
ICP spectroscopy/ flame AAS	metals	90	110
Furnace AAS	metals	90	110
Cold vapor AAS	mercury	80	120
Other	cyanide	85	115

A quarterly linear range verification check standard is analyzed for each element. The analytically determined concentration of the standard must be within 5% of the value. This concentration, then, is the upper limit of the ICP linear range. Results cannot be reported beyond that upper concentration level unless they are a result of an appropriate dilution/reanalysis. At the beginning and end of each ICP analysis shift, ICP interference check samples are analyzed. This verifies interelement and background correction factors since it assesses analytes of interest in the presence of high concentrations of other interfering elements. Results must fall within ±20% of the true value.

For each batch of samples processed, an ICP serial dilution analysis is performed. If an analyte is present at a sufficient concentration (minimally a factor of 50 above the instrument detection limit [IDL]), an analysis of a 1:4 dilution must agree within 10% of the original determination. If not within that limit, a chemical or physical interference effect is likely, and the associated data are qualified.

All QC sample results are tabulated immediately following analysis and compared to the contract-mandated, method-mandated, or client-mandated control limits for precision and accuracy. Out-of-control results are cause for immediate repreparation and/or reanalysis. No outlying data are ever released until the laboratory has verified that unacceptable results are attributable to the sample matrix.

Instrument detection limits are determined quarterly for each ICP and AA spectroscopy system used for inorganic analyses. This is accomplished by multiplying by three, the average of the standard deviations obtained on three nonconsecutive days, from the analysis of a standard of each analyte in reagent water. The concentration of each analyte in the standard solution is at approximately 3-5 times the estimated instrument detection limit and seven consecutive measurements, per day, per analyte, are required.

Also, interelement correction factors are determined annually for ICP analysis. This determines the potential false analyte signals caused by commonly occurring high levels of elements found in environmental samples. Correction factors for spectral interferences are reported for all ICP instruments at all wavelengths used. For more detailed information on calibration procedures used in the Inorganics Laboratory, refer to Section 8.1, Tables 8-5 through 8-6.

12.0 Performance and System / Quality Assurance Audits

The QA staff conduct two types of internal audits: system audits and performance audits. A system audit is an on-site inspection or self-assessment of the laboratory's control system. While performance audits are a quantitative appraisal, system audits are more qualitative in nature, intended to provide evidence of the laboratory's competence. The auditor applies specific audit methods, evaluates audit findings, and reports these findings to laboratory management. The auditor conducts a follow-up review at a later date to verify that management has acted on these findings to improve processes.

The laboratory has developed a Total Quality Management program, dedicated to improving the quality of all parts of the system. The QA staff are assigned individual departments with total auditing responsibilities. The QA auditor conducts audits of these individual areas, with at least one general system audit each quarter for each area. Performance audits are conducted continuously.

12.1 Quality Assurance Audit Unit

The QA specialists and QA manager conduct ongoing routine system audits. This unit functions independently from laboratory operations and reports to the Vice President General Manager (VPGM), who reports to the CEO. The QA department staff consists of senior scientists with bachelor of science degrees in chemistry or other applied science, five or more years of environmental analytical laboratory experience, and at least two years of experience using laboratory QA/QC techniques and basic statistical principles. The QA Specialist I position (there are two more advanced specialist levels) requires a minimum of two years in GC, GC/MS, or inorganics sample and data analysis.

QA Specialist I training begins with gaining a comprehensive understanding of the laboratory QA program, QA Program Plan, and SOPs; the organization of the laboratory; sample and data flow; sample tracking and scheduling through the LIMS; analytical and non-analytical laboratory SOPs; and good laboratory practices. Training materials have been prepared to facilitate QA specialist training.

Senior QA staff and management train QA specialists in auditing principles by allowing them to observe, and by accompanying them on internal and external system audits. After at least five training audits, the QA specialist demonstrates proficiency in auditing techniques by coordinating and participating in an onsite audit of their assigned area.

12.2 System Audits

System audits are performed both by internal and external auditors. The QA department performs internal system audits. Commercial clients and federal and state certifying agencies perform external system audits. A system audit is performed to qualitatively assess the laboratory's control system and is intended to provide evidence of the laboratory's competence.

The objectives of a system audit include ensuring that

- management is committed to creating a work environment dedicated to quality and that a structured management system is in place to support an effective QA program,
- the QA program is documented and implemented to assess work to ensure technical, administrative, and quality objectives,
- personnel are adequately trained and qualified to do their jobs,
- senior management regularly assesses the effectiveness of management controls and the adequacy of resources available to achieve and assure quality,
- procured items and services meet established requirements and perform adequately,
- procedures are established and maintained for the timely preparation, issuance, control, and revision of documents, including documentation of review and approval, and that records are specified, prepared, reviewed, approved, and maintained for evidentiary purposes,
- computer hardware meets requirements and that any changes are controlled; computer software is developed, validated, verified, and documented; and that any changes are controlled,
- work performed complies with established technical standards and administrative controls, as well as safety policies, and
- procedures are established for detecting and preventing quality problems and for ensuring quality improvement.

Internal System Audits

The following quarterly internal audit functions are performed:

Good laboratory practices for logbooks / recordkeeping -- Verifies that proper recordkeeping practices are being followed according to those defined in QA SOPs and that logbooks are current and complete, with documentation of supervisor review.

Data storage/archival/document control -- Verifies that data are stored and controlled properly, that COC procedures are followed, and that sample activity is traceable.

Sample storage/COC -- Verifies that samples are stored and controlled properly, that COC procedures are followed, and that sample activity is traceable.

SOP compliance -- Verifies by observation that procedures are being followed according to written SOPs. Also verifies that written SOPs are compliant with the methods upon which they are based.

Reagent/standard storage control -- Verifies that reagents, standards, and other chemicals are stored and controlled properly.

Standards preparation/traceability -- Verifies that new standard lots are properly tested against previously approved lots. Percent deviation criteria must be met for new standard lots to be accepted.

Cooler alarm test -- Verifies that cooler alarm system is functioning correctly with proper notification of excursions.

Routine checks of QC test samples -- These are performed regularly and include checks of vendor-supplied glassware, glassware decontamination, water purification, refrigerated storage system, and reagent purity.

Data audits -- These are performed regularly, and the objective of these audits is to look at 5-10% of data packages generated, after they have been submitted to the client. During these audits, QA staff verify accuracy, completeness, and usability of the data.

Customer Problem Resolution Report (CPRR) follow-up -- Verifies that corrective actions have taken place, are still in effect, and that nonconformance recurrence control is active.

Internal audit follow-up -- Verifies that nonconformances or problems noted during an internal audit have been corrected.

External audit follow-up -- Verifies that nonconformances or problems identified in an audit report from external auditors have been corrected or have specific target completion dates for corrective action.

The following semiannual internal system audit functions are performed:

Facilities/maintenance schedule -- Verifies that facilities and equipment are adequate and properly maintained and that laboratory areas are free from interferences or contaminants.

Warehouse audit -- Verifies that all warehouse operations are functioning properly.

GC/MS tape audit -- Involves checking GC/MS tapes for adherence to contractual requirements and to ensure the consistency of data reported on hardcopy/diskettes with that generated on GC/MS tapes.

The following internal system audit functions are performed annually:

Subcontract laboratory audits -- Consists of performing a system audit of all QA-approved subcontract laboratories.

Training documentation/qualifications -- Verifies that training records are current and that personnel meet the requirements stated in the current U.S. EPA CLP SOW or method as well as those specified by individual states.

Vendor audits -- Verifies that procured items and services that directly affect the quality of results or products conform to established specifications.

Good automated laboratory practices/software validation -- Verifies that good automated laboratory practice guidelines are being followed and that all software systems have been properly validated.

The system auditor should be accompanied by area management so that both can observe the operation first hand from a QA/QC perspective, and can constructively discuss administrative or operational problems encountered when they are observed. This gives laboratory management an opportunity to clarify or correct potential misunderstandings so that observations are not inaccurately described in a report to senior or executive management.

If deficiencies/nonconformances are observed during the system audit, a CPRR may be initiated by the auditor. An audit report, which describes the nonconformances is distributed to the management of the audited areas. Management is required to respond to the report in writing. Corrective actions to remedy deficiencies or nonconformances noted in the report are verified in follow-up audits on a quarterly basis.

Corporate Quality Management Systems Audits

All of the above QA department auditing activities are considered in assessing the overall quality management systems. QA department auditing activities are summarized by the QA manager and reported quarterly to the VPGM. Based on this report, the VPGM assesses the integrated quality assurance program and its performance in a quarterly report to the CEO and senior management. These assessments focus on how well the QA program is working and identify management problems that hinder the organization in achieving its objectives in accordance with quality requirements. The effectiveness of the system of management controls established to achieve and assure quality is evaluated along with the adequacy of resources and personnel. Senior management take prompt action in response to the quarterly assessment and document any resulting decisions. Follow-up by the VPGM includes an evaluation of the effectiveness of management's actions.

External System Audits

CompuChem is also audited extensively by external agencies, contractors, and third parties. Certification officers from various state agencies, including North Carolina,

California, New Jersey, New York, Wisconsin, Florida, Massachusetts, South Carolina, Connecticut, and New Hampshire, conduct system audits of the laboratory. Most of these state certification programs specify that on-site inspections are to be conducted annually. CompuChem is also audited by representatives of the Navy Energy and Environmental Support Activity (NEESA), the Hazardous Waste Remedial Actions Program (HAZWRAP), and the Army Corps of Engineers. As a CLP contractor, the laboratory is also audited by the U.S. EPA (usually the regional office, but often including administrative project officers from headquarters) and technical and evidentiary auditors contracted by the CLP. Additionally, many clients conduct inspections or hire third party QA auditors to inspect the laboratory before start-up and during the course of larger, more critical, or politically or legally sensitive projects.

CompuChem requests notice at least two weeks before a scheduled audit to ensure that management and QA staff are available. However, an external audit may be conducted (announced or unannounced) at any time during normal business hours. To protect client confidentiality, some documents (particularly those identifying clients, sites, or projects) will not be made available for inspection except to those directly involved in such projects or authorized state or federal officials or authorized third parties.

Any deficiencies/nonconformances observed by the auditors are included in an audit report that is generally written by a QA staff member within a week of the visit. CPRRs are initiated, as required, in response to the audit findings. When the final audit report is received from the auditors, it is compared with the QA audit report to see if any additional findings have been noted that need to be addressed. Once the responses to the CPRRs are completed, a formal response is compiled by a QA staff member and submitted to the external agency as required. Copies of the response are circulated to internal management.

12.3 Performance Audits

The QA staff conduct performance audits to evaluate the quality of the data produced by the analytical system. These audits are performed independently of and in addition to routine QC checks, and reflect as closely as possible laboratory performance under normal operating conditions. Often, as a result of deficiencies observed during ongoing performance audits, a full system audit may be initiated.

Internal Performance Audits

Internal performance audits include analysis and assessment of double-blind performance evaluation (PE) samples and single-blind QC reference standards, assessment of proficiency tests for new methods, assessment of method detection limit and method validation studies, and assessment of QC repeat statistics.

Double-blind PE samples are ordered from a certified outside source and packaged into a SampleSaver. They are received in the laboratory under a dummy account number (blind even to the receiving clerk) and processed in exactly the same manner as a field sample. The data report is mailed to a cooperative laboratory and reported back to CompuChem's QA auditor. Only the QA auditor and marketing representative are aware of the introduction of the PE sample into the system. Double-blind PEs are introduced at least semiannually. The selection of parameters and methods is largely based on laboratory performance on external PE samples and on results of

system audits. Results are summarized in a report to management and corrective actions are taken as necessary.

Single-blind QC reference samples are analyzed concurrently with most external PE samples, at least quarterly. These samples are prepared from NIST- or EPA-traceable reference materials, when available, and are used as additional evidence that analytical systems are in control at the time of analysis. These results are filed for historical performance review and trend analysis as needed.

Control charting is performed for all laboratory methods and matrices. Laboratory control sample (LCS) recoveries are plotted daily and any out-of-control conditions are flagged by the software program. The control charts are evaluated daily by laboratory staff. The QA staff audits control charts and evaluations monthly.

Method detection limit (MDL) studies are performed annually and are reviewed and approved by the QA department. Method validation studies are performed as part of the development process for new methods and are reviewed and assessed by the QA department. QC repeat statistics are summarized by the QA manager in the QA monthly report.

External Performance Audits

CompuChem also participates in a number of external, interlaboratory PE studies; one to four external PE studies are conducted each month throughout the year. These include the Water Pollution studies originating from EPA-Cincinnati, the NYDOH non-potable PE series, the EMSL-LV radiological PE studies, the DOE-EML radiological intercomparison studies, samples from state certifying agencies, and independent PE studies to support the HAZWRAP/NEESA/DOE and the Army Corps of Engineers programs.

As a participant in the U.S. EPA CLP, the laboratory is also required to successfully analyze quarterly, blind proficiency samples for both organic and inorganic parameters. The CLP provides reports comparing laboratory performance with all other contract laboratories in the CLP, in addition to (approximately quarterly) Laboratory Profile Packages summarizing laboratory performance for routine QC parameters (surrogate and spike recoveries, RPDs, turnaround time, etc.). U.S. EPA Region IV also submits double blind spiked samples with each SDG and reports the results back to the laboratory with corrective action requirements specified for any failing parameters.

Results from other external PE samples are summarized and reported to senior management by a designated QA staff member. CPRRs are issued as required for any deficiencies found in the PE sample results. Responses to deficiencies are required from the appropriate laboratory area manager or his/her designee. A formal response is then compiled by the QA staff member and submitted to the external agency as required.

13.0 Equipment and Instrument Maintenance

CompuChem is a high-capacity laboratory that maintains a large inventory of instrumentation that facilitates rapid sample turnaround time. Expert technicians maintain and service the instruments, which reduces the possibility of time lost due to instrument failure. Historically, instruments have been properly functioning 97% of the time. Alternative instruments are always available. CompuChem has 10 GC/MS instruments dedicated to analysis of volatile compounds in samples, and 12 GC/MS instruments dedicated to analysis of semivolatile compounds in samples. These two sets of GC/MS instruments are physically separated from one another to prevent cross-contamination. Instruments within each of the two sets are arranged in working clusters for maximum efficiency, and can be reconfigured as needed.

Full-time experts maintain the analytical instruments, and perform routine and preventive maintenance and major instrument repairs on site. The instrument repair experts have a large in-house stock of spare parts to expedite repairs. CompuChem also has service agreements with instrument manufacturers to further support the instrument maintenance and repair program. Keeping instruments operational at all times is the key to CompuChem's prompt completion of routine analytical tasks governed by demanding programmatic requirements such as those of the U.S. EPA's CLP.

13.1 Hardware Tuning and Calibration

Tuning and calibration of instruments are documented in the instrument runlogs at the bench. If an instrument fails tuning or calibration criteria (see Section 8.0), hardware adjustments or other appropriate maintenance is performed and documented, and the analyst repeats the tuning and calibration attempt. If the second attempt is successful, this is entered into the runlog and sample analysis may proceed. If the second attempt is not successful, corrective actions may include additional maintenance by the full-time instrument service technicians in our instrument support group.

Sample analyses may not proceed without an acceptable calibration. Any equipment that cannot be successfully calibrated and returned to service before the next working shift is clearly labeled *OUT OF SERVICE. DO NOT USE*. The fact that the calibration was unsuccessful is recorded in the instrument runlog, if a runlog is used. If the equipment does not require a runlog (e.g., analytical balance), a sign is affixed to the equipment until the equipment is recalibrated and returned to service.

Instrument maintenance services at CompuChem differ for GC/MS instruments and other hardware. CompuChem staff have full maintenance and repair responsibilities for GC/MS instruments, and have been formally trained by the instrument manufacturer or other qualified instrument service organization. Instrumentation support staff document instrument repair on a service report. Historical activity records for each instrument are kept on file.

Although most maintenance is performed in house by senior chemists or instrumentation support staff, some instruments and complex repairs require that maintenance be performed by the instrument manufacturer or supplier. For this reason, service contracts are in place that include periodic maintenance by the vendor, although maintenance personnel initially assess

the instrument problem to determine whether repairs can be performed in house.

Qualified senior analysts must be able to perform routine preventive maintenance on instruments; learning to do this is part of each senior analyst's training program. Manufacturer's manuals for each instrument are present in the laboratory in which the instrument resides. These manuals include procedures for instrument calibration and for performing routine and preventive maintenance. Hardcopy SOPs are in place that describe these activities step-by-step.

Preventive maintenance requirements for all laboratory instrumentation and equipment are described in Tables 13-1 through 13-12. Instrument maintenance is recorded in standardized, permanently bound laboratory logbooks that are subject to strict document control enforced by the Technical Communications department.

13.2 GC/MS Instrumentation

Preventive maintenance checks and services required for GC/MS instrumentation are presented in Tables 13-3 through 13-7. Instrumentation support staff perform these services every three months. Service records are retained permanently. The instrument operator performs routine preventive maintenance every 12 hours or as needed (Table 13-7). This service is documented in individual instrument runlogs, which are bound and archived according to the procedures described in Section 9.0. The instrument operator completes a service record for non-routine service performed by the instrument support staff.

13.3 GC Instrumentation

Most service on GC instruments is also performed in house, with the exception of non-routine hardware maintenance and some computer board malfunction repairs. Preventive maintenance in the GC Laboratory follows a set schedule, and records of preventive maintenance are kept at each instrument. Records of non-routine service are kept by the instrumentation support staff.

13.4 Inorganics Laboratory and Organic Characterization Laboratory Instrumentation

Service contracts for instruments in the Inorganics Laboratory and in the Organic Characterization Laboratory have been purchased, and records of services performed are kept at each instrument. Routine preventive maintenance is performed according to the schedule set forth in Table 13-10.

13.5 Radiological Laboratory Instrumentation

All preventive maintenance and service records are kept by the Radiological Laboratory manager. Frequency of preventive maintenance is shown in Table 13-12. All instruments in this laboratory are under service contract, and service engineers inspect the units at least annually to determine whether additional maintenance may be necessary.

13.6 Service Contractors

Any repair performed by a service contractor is documented by attaching copies of the work order to the instrument maintenance logbooks within each laboratory. When logbooks are filled, they are submitted to the Technical Communications department for archival.

13.7 Equipment Monitoring

Balances, ovens, and refrigerators are monitored regularly (Table 13-1). The analysts use standardized logbooks to record thermometer readings and daily balance calibrations. Facilities support staff keep records of inspection activities and affix adhesive inspection labels with dates of inspections of equipment such as ventilation hoods and fire extinguishers.

In case of other equipment failure, most systems have built-in redundancy features. The computer systems, including mainframes, have redundant software and programming stored on back-up shadow systems. All instruments have multiple redundant systems available, except for the FT-IR instrument and the TOC and TOX analyzers. Graphite furnace AA instruments may be used to perform most trace metals analyses if either ICP instrument is disabled. For the other exceptions, approved subcontractor laboratories are available to perform analyses on short notice in such emergencies. In such a case, CompuChem notifies the client and gains the client's approval before allowing a subcontractor laboratory to perform analyses.

Table 13-1. Equipment Monitoring

Equipment	Check	Acceptance Criteria	Frequency
top-loading balances	Verify accuracy with weights calibrated against certified class-S weights	±0.05 g	daily or with each use
analytical balances	Verify accuracy with certified class-S weight using different weight each day over range of weights routinely determined. Calibrated by outside source quarterly.	±0.00005	daily or with each use
thermometers	Verify accuracy against NIST-certified thermometer, or if purchased with certificate of traceability, check for mercury separation daily.	±0.1 °C	annually
temporary storage refrigerators	Verify temperature within range.	4°C ± 2°C	daily
long-term storage refrigerators	Verify temperature within range.	4°C ± 2°C	twice/day (8 AM, 5 PM)
freezers	Verify temperature within range.	-10 to -25°C	daily
water baths	Verify temperature within range.	60-80°C 80-90°C	daily
ovens	Verify temperature within range.	105 ± 5°C	daily or with each use
fume hoods	Check fan condition and velocity.* Class A: 125-150 cfm Class B: 100 cfm half sash open 80 cfm (full) Class C: 75-80 cfm half sash, 50-60 cfm (full)		monthly
safety showers	Inspect for working order.	NA	quarterly
fire extinguishers	Pull pin in place.	gauge reads full	monthly
eyewash stations	Inspect for working order.	NA	quarterly

*Class A hood is used for extremely toxic or hazardous materials. Class B hood is used for common lab chemicals and volatile solvents of average toxicity. (Most hoods in the laboratory are Class B hoods.) Class C hood is used for low toxicity chemicals and solvents such as acetone, methanol, ethanol, and other hydrocarbons.

Table 13-2. Preventive Maintenance Checks and Services: Gas Chromatograph

Items to be Inspected	Probable Problem	Service Interval	Procedure (internal)*
line fuses (GC)	inactive GC; blown fuse	as required	replace fuse
injector for packed columns heater or sensor	failure to heat	as required	replace heater or sensor
splitless injector for capillary columns heater or sensor	failure to heat	as required	replace heater or sensor
injector septum in the GC	obstruction/leaks	daily	clean, inspect, or replace as required
injector liner	poor chromatography	as required	clean, inspect, or replace as required
carrier gas connections and couplings	leakage	as required	tighten or replace fittings
carrier gas filter in the GC	obstructed; low flow rate	as required	replace when new gas cylinder installed
Filter flow controller	dirty filter	every 3 months	replace filter
capillary column	poor chromatography	as required	inspect or replace as needed
packed column (glass)	excessive usage, leaks at injection and interface port of the zone-heating block	as required	inspect or replace as needed
packed column (metal)	excessive usage, leaks at injection and interface port of the zone-heating block	as required	inspect or replace as needed
detector heater	GC not ready	as required	replace heater or sensor
GC cooling fan	blown fuse	daily	inspect or replace fuse or fan as needed

*Applicable procedures are presented in the Finnigan operator manual.

Table 13-3. Preventive Maintenance Checks and Services: Mass Spectrometer

Items to be Inspected	Probable Problem	Service Interval	Procedure (internal)
glass jet separator	obstruction or glass breakage	as required	clean or replace
glass jet separator ferrules	obstruction or glass breakage	as required	replace
mass analyzer head assembly (in the vacuum manifold)	gross leaks, persistent pressure due to degassing of trapped gases in the vacuum system, faulty CAL gas pressure, faulty switch	as required	inspect
quadrapole mass analyzer	failure to pass tune; overuse	every three months	inspect or replace
electron multiplier	low sensitivity	as required	inspect or replace
Alcatel vacuum pumps	locks up	every three months	purge weekly and replace oil
Pfeiffer turbo pump	dirty oil	weekly	purge weekly and replace oil
Seizer turbo pump	dirty oil	every three months	purge and replace oil
vacuum system filter	excessive use, dirty filter	as required	clean and inspect
ion source	lack of sensitivity, irregular peak shape, no autotune	every three months	clean, inspect, or replace

Table 13-4. Preventive Maintenance Checks and Services: GC/MS Interface Oven

Items to be Inspected	Probable Problem	Service Interval	Procedure (internal)
capillary interface tubing	plugged	as required	clean, inspect, or replace
separator divert fitting	leakage	every three months	tighten or replace
vacuum divert valve	clogged	every three months	clean, inspect, or replace

Table 13-5. Preventive Maintenance Checks and Services: GC/MS Power Module

Items to be Inspected	Probable Problem	Service Interval	Procedure (internal)
MS power supply	low or missing voltage	every three months	measure and verify printed circuit board (PCB)
turbo power supply	failure to function	every three months	measure and verify PCB, or replace

Table 13-6. Preventive Maintenance Checks and Services: GC/MS Card Cage Module

Items to be Inspected	Probable Problem	Service Interval	Procedure (internal)
air filter at bottom of cage	dirty filter, obstruction of air flow	every three months	clean
fan	burned out fan	every three months	clean
signal cable on digital I/O PCB	no signal	as required	inspect for secure fit or replace

Table 13-7. Preventive Maintenance Checks and Services: Nova Computer

Items to be Inspected	Probable Problem	Service Interval	Procedure (internal)*
fan	faulty fan rotation	as required	inspect or replace
output signal	failure to boot	as required	check and verify
adjustable DC	low voltage	as required	check and verify
disk drive	crash	as required	inspect and replace check software

Table 13-8. Routine Preventive Maintenance: Inorganics Laboratory Instrumentation

Instrument	Routine Maintenance Performed	Frequency
AA	Check and clean air filter.	daily
	Check cuvettes.	daily
	Check IR sensor window.	daily
	Check and replace marble chips.	daily
	Check drain lines.	daily
ICP	Clean torch.	as required, minimum weekly
	Check and clean filters.	as required, minimum weekly
	Clean nebulizer chamber area.	as required, minimum weekly
	Replace pump tubing	as required, minimum weekly
TrAAcs 800	Wash reagent lines.	daily
	Replace worn reagent tubing lines.	as needed; checked daily
Technicon autoanalyzer	Check and replace worn pump.	daily
	Clean phase separator.	weekly
Lachat	Wash reagent lines. Replace worn reagent tubing lines.	daily as needed; checked daily
Leeman	Change pump tubings.	weekly
	Change reductant tubing and sample and mixing coil tubing.	every 2-3 months
	Change sample probe.	as needed
	Replace optical cell.	monthly

Table 13-9. Routine Preventive Maintenance: GC/MS Laboratory Instrumentation

Instrument	Routine Maintenance Performed	Frequency
Volatile GC/MSs	Change septum, column maintenance, clean injection port liner, back flush purge and trap device, change trap.	as needed; checked each shift
Semivolatiles GC/MSs	Change septum, clean or replace injection port liner, clean injection port, perform column maintenance.	every 12 hours

Table 13-10. Routine Preventive Maintenance: Organic Characterization Laboratory Instrumentation

Instrument	Routine Maintenance Performed	Frequency
total organic carbon Dohrmann DC-180 (with boat sampler)	Change catalyst with sludge/sediment.	monthly
	Clean sludge/sediment sampler.	daily
	Check pH of gas/liquid separator (pH <2).	monthly
	Change pump tubing.	monthly
	Check gas flow rate.	daily
	Change copper and tin in scrubber.	semiannually
	Clean dust out of electronics cabinet.	every three months
	Clean sample cell on IR detector	semiannually
	Empty water trap.	daily
total organic halides Dohrmann DX-20A	Clean combustion tube and filtration cell with chromic acid.	daily
	Clean sampling path.	daily
	Verify proper gas flow rates.	monthly
	Clean dust out of electronics cabinet.	monthly
FT-IR Nicolet Model 42	Test sequence to verify laser, detector (source), nitrogen, and scan are working.	daily

Table 13-11. Routine Preventive Maintenance: GC Laboratory Instrumentation

Instrument	Routine Maintenance Performed	Frequency
GC/ECD	Replace septum.	every 72 hours
	Replace first 2-3 inches of column packing.	every 72 hours
	Replace injection port liner.	every 72 hours
	Swab injector port with a series of four solvents.	every 72 hours
GC/VOA	Replace ELCD detector solvent.	weekly
	Replace roughening resin.	semiannually
	Replace finishing resin.	annually
HPLC	Column flushed with methanol.	daily

Table 13-12. Routine Preventive Maintenance: Radiological Laboratory Instrumentation

Instrument	Routine Maintenance Performed	Frequency
Packard liquid scintillator	None required except inspect sample changers.	weekly
Tennelec Low Level Alpha/Beta	none required	NA
Eberline radiation monitor	Clean as needed.	at least weekly
EG & G gamma detector	Make sure there is an ample supply of liquid nitrogen.	weekly
NMC radon counter	Check for dust and clean.	at least weekly
Ortec alpha spectrometer	none required	NA

14.0 Quality Assurance Objectives for Measurement

To meet the objectives of the QA program as they are described in the QA policy statement (Section 3.0), senior management supports a program designed to:

- assess the capabilities of analytical methods for meeting users' needs in terms of accuracy, precision, completeness, representativeness, and comparability;
- establish and monitor the routine operational performance of our laboratory through appropriate systems checks to ensure that all aspects of the QA program are operative; and
- assure that corrective actions are taken and that system control has been restored before resuming sample analysis.

The first of these processes will be discussed in this section. Section 11.0 describes the laboratory's methods for fulfilling routine QC requirements. Auditing programs, proficiency testing programs, and corrective action procedures are discussed in Section 12.0.

Following is an overview of CompuChem's QA objectives for precision, accuracy, respresentativeness, comparability, and completeness.

14.1 Precision

The laboratory objective for precision is to meet or exceed the method-specified or client-specified precision requirements as applied to samples of similar matrix and concentration. To evaluate precision between matrix spike duplicates and inorganics sample duplicates relative percent difference (RPD) criteria published by the U.S. EPA for its CLP SOWs for inorganic and organic analyses, and those determined from internal laboratory performance data, are used . The formula for determining RPD is:

$$\frac{|MS - MSD|}{1/2\,(MS + MSD)} \times 100 = \text{Relative Percent Difference}$$

MS = spike recovery for matrix spike
MSD = spike recovery for matrix spike duplicates

$$\frac{|S - SD|}{1/2\,(S + SD)} \times 100 = \text{Relative Percent Difference}$$

S = sample analyte concentration
SD = sample duplicate analyte concentrate

14.2 Accuracy

The laboratory objective for accuracy is to meet or exceed the accuracy requirements dictated by the method, contract, or client, and as applied to samples of similar matrix and concentration. To evaluate accuracy in matrix spike, matrix spike duplicate, and blank spike QC samples, CompuChem uses percent recovery criteria published by the U.S. EPA in their CLP SOWs for inorganic and organic analysis, those published in the *Federal Register* (40 CFR 136, October 26, 1984), and those determined from internal laboratory performance data. The equation used to determine accuracy is:

$$\frac{MS - US}{S} \times 100 = \text{Percent Recovery}$$

MS = concentration of target analyte in spiked sample
US = concentration of target analyte in unspiked sample
S = spiked standard concentration

14.3 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. CompuChem's objective for completeness is to provide unqualified data of the highest quality for 100% of samples. Factors beyond the laboratory's control that adversely affect attainment of completeness objectives include:

- receipt of samples in broken containers
- receipt of samples whose COC or sample integrity is compromised in some way
- receipt of samples with insufficient volume to perform initial analyses or repeat analyses should initial efforts not meet QC acceptance criteria
- receipt of improperly preserved samples
- receipt of samples held in the field longer than expected so that holding time requirements are jeopardized
- receipt of incomplete or inaccurate information resulting in the application of incorrect methods
- assessment of sample data by end-users using criteria other than those stated in applicable method references or applicable data validation guidelines

When requested, the completeness of an analysis can be documented by including in the test report sufficient information to allow the data user to assess the quality of the results. This information may include such items as chromatograms, mass spectra, a summary of QC sample results, and the tabulated analytical results.

Additional results, up to and including all data sufficient to recreate the entire analyti-

cal process, are optional deliverable items. These may include laboratory worksheets, calibration data, all QC sample data, and internal COC documents. The highest level document emulates that required under the U.S. EPA CLP, and is intended as a legally defensible document in itself. The raw data (before data reduction) are archived indefinitely on magnetic tape and stored in a secured area within the facility.

14.4 Representativeness

Representativeness is a qualitative characteristic, and is considered a goal to be achieved rather than a quantitative measurement. It describes the degree to which the analytical results of a particular sample accurately and precisely represent results characteristic of other samples taken from the same site during the sampling event. Representativeness is dependent upon both the sampling program design and proper laboratory protocol.

For many sample types, true representativeness can be accomplished by careful collection planning, sample compositing, and/or sample splitting. Soil samples and samples of complex or heterogeneous matrix usually present the greatest difficulties for samplers and analysts alike. The sampler should make every effort to homogenize the sample during collection. Laboratory technicians must, whenever practical, homogenize or thoroughly mix the sample before removing aliquots for analysis. CompuChem's sample preparation SOPs include specific procedures for compositing and homogenizing as-received samples. Representativeness can be assessed by analyzing field duplicate samples.

The manner in which the data are correlated to the particular sampling episode and sample site(s) are major considerations when evaluating representativeness. When the laboratory is aware of conditions adversely affecting data representativeness, a QA Notice or Laboratory Notice is included in data packages to qualify results and to provide guidance in interpreting data usability.

14.5 Comparability

Comparability refers to the confidence with which one data set can be compared with another. The analytical results can be compared to results of other laboratories because the objectives of the laboratory for comparability are to:

- demonstrate traceability of analytical/calibration standards to NIST, EPA, or other certified sources
- use standard methodology
- adhere to instrument tuning and calibration procedures and frequency requirements
- apply appropriate levels of QC within the context of the QA program
- participate in interlaboratory studies and independent proficiency testing programs to document laboratory performance

By using traceable standards and standard methods, the analytical results can be compared to other laboratories operating similarly. The QA program documents QC performance and the interlaboratory studies document performance compared to other laboratories. Additionally, internal quarterly blind proficiency studies are instituted as a means of monitoring intralaboratory performance.

14.6 Data Quality Objectives

Data quality objectives (DQOs) are used in planning environmental data collection activities. They are meaningful because meeting these objectives assures that the data will support the decision. They establish the level of data that will support the decision. They establish the level of uncertainty in results that a decision maker is willing to accept. They can be used to define QA/QC programs specific to a project or data collection activity. DQOs have been established for programs under which the laboratory provides analytical services. Internal project support staff work closely with the client and regulatory agencies to ensure that DQOs will be met by the analytical results provided. The project management teams at CompuChem convey project- and client-specific requirements to the laboratory by using and distributing project profile sheets (PPS) and holding weekly team meetings.

15.0 Corrective Action and Documentation

When out-of-control events occur, they are documented and corrective action is implemented. How such events are documented depends on the type of event and area in which the out-of-control event occurs. Most out-of-control events identified during routine sample processing are restricted to single samples, batches, or data reports. Some examples of these types of events include:

- incorrect client identifier on COC documents
- broken or unpreserved samples received
- lab accident during sample processing
- accuracy failures for surrogate and/or spike standards
- precision failures between duplicates
- variations in internal standard responses
- contamination of method blank
- errors or omissions in data reports

The types of excursions that occur and the corrective action documentation that results during different stages of sample processing are discussed in the following section.

15.1 Discrepancies Noted During Sample Receipt

Any discrepancies noted during the sample receiving process are documented on the COC form. Information about discrepancies is provided to the customer service representative, who is responsible for contacting the client about the problem and documenting the resolution in a phone log. If the client instructs the laboratory to proceed with sample analysis, a QA Notice explaining the problem is included with the data package. This process is commonly used when samples are received at higher than required temperatures or have been improperly preserved. If a sample is broken during transport to the laboratory, the customer service representative notifies the client, who, in turn, notifies the field crew to resample if possible. The client also instructs the laboratory on the disposition of the received sample.

15.2 Out-of-Control Events at the Bench

Out-of-control events noted and corrected at the bench by technicians or analysts and proof of return to control are documented on sample preparation worksheets, sample analysis worksheets, instrument runlogs, calibration or temperature logbooks, or instrument maintenance logbooks, as applicable. These forms may serve as documentation of action taken as a result of a failure. When a failure occurs, the analyst records a comment in the appropriate field of this report and notifies the Production Planning and Control department to reschedule the sample for the process that must be repeated. The analyst is required to document the failure and required corrective action by using the comments field, signature, and condition code, if applicable. Condition

codes are recorded on the report as well as entered in the LIMS in the appropriate analytical field, i.e., sample preparation procedure queue. Condition codes, further described in Section 16.0, are used to describe the status of a sample analysis and whether reanalysis is required. The codes allow for trend analysis to be presented in a series of tables and graphs in the QA department's monthly report to management.

15.3 Errors or Omissions in Data Reports

If errors or omissions in data reports are found during any of the review processes, a Go-Back form is completed and the data are returned to the appro priate area for correction. The actual correction made and the person who made the correction are documented on the Go-Back form. A QA Notice or laboratory notice is included with any reported data that do not meet all QC criteria or which require explanation. This notice explains any out-of-control events and corrective actions taken to remedy them.

Observations documented in these notices are also summarized in the case narrative, which is included with most styles of data reports. Case narratives are written by the final technical reviewer of the data package, who authorizes release of data package.

15.4 Out-of-Control Events Observed During QA, Management, or External Audits

During various internal or external audits, an out-of-control event might be observed by QA, management, or external auditors that can adversely affect laboratory quality on a more global basis. These events might affect, for example, entire analytical systems; projects; data usability or integrity; or sample integrity, security, or safety.

An unacceptable result on a performance evaluation (PE) study or internal blind PE study can also be an indication of a systematic problem. Other examples include:

- temperature excursions on successive days in a sample storage refrigerator
- improperly calibrated analytical equipment
- poor spike recoveries on multiple extractions of an LCS
- contaminated glassware or sample/standard storage
- inaccurate/incomplete SOP or personnel not following SOP
- deviations in COC documentation or procedure
- obliterations, writeovers, or other improper data corrections
- expired standards on the "active" shelf of a refrigerator
- open or unlabeled waste containers
- inadequate hood velocity

- recurring out-of-control conditions evident on control charts

These out-of-control events and the corrective action taken to resolve them are documented in several ways, such as:

- An interoffice memorandum is written to the responsible party describing the out-of-control event and requiring a written response including corrective action by a specified date.
- After internal or external audits, a formal report is written to all involved laboratory areas that describes any nonconformances or deficiencies found. Written responses from the responsible parties, which detail corrective actions take, are required. For external audits, these responses are included in the formal written response to the auditor.

Quarterly follow-up audits are performed by the QA department to ensure that corrective actions have been implemented . These audits are documented on forms and filed in binders in the QA department office.

A CPRR (see Figure 15-1) is issued and completed when an external or internal client identifies a deficiency. This form documents what the out-of-control situation is, who is responsible for correcting the problem, what action is to be taken, and the target date for implementation.

The nonconformance is closed once QA or lab management has determined that implementation is complete and that system control is restored. These reports are kept on file by the coordinator and can be used for trend analysis. Control chart evaluation forms are completed by the laboratory to document the assessment of out-of-control statistical events that occur during sample processing. Any necessary corrective actions are noted on the forms. The QA department audits the evaluation forms monthly.

Tables 15-1 through 15-4 summarize QC sample frequency, acceptance criteria, and corrective action in all laboratory areas for method blanks, blank spikes and LCSs, matrix spikes and MSD pairs, and duplicates. Acceptance criteria limits for CLP methods are defined by the published SOW. Limits for non-CLP methods or limits that are not specified in the method are subject to change based upon updated intralaboratory statistical performance data. Generally, acceptance criteria are advisory unless the client requires specific corrective action steps for failures.

Figure 15-1

COMPUCHEM ENVIRONMENTAL CORPORATION
CUSTOMER PROBLEM RESOLUTION REPORT

ORIGINATOR (TO BE COMPLETED BY ORIGINATOR)

Client Name/Project _____
Address _____
Address _____ Phone # _____
Priority _____ Account # _____
Case # _____ SDG # _____
Rpt. Style _____ Order # _____

Date inquiry rcvd _____ Internal? Y or N
Originator/AA/Sales Rep _____
Desired response date _____
Priority Code
CPR Codes

CC #s

Attachments
_____Order _____Req _____Phonelog
_____Quote _____Note _____Other

Notes/Problems/Background _____

RESOLUTION (TO BE COMPLETED BY ASSIGNEE)

Date rcvd _____ Referred to _____ CPR Contact date _____
Commit date _____ Client issue valid? Y or N Contact with Originator _____
Completed date ____ Resolved by _____ New Codes
Resolution time _____

Notes/Action/Info _____

QA/CORRECTIVE ACTION/NON-CONFORMANCE RECURRENCE CONTROL

Corrective Action Required? Y or N Target completion date _____
QA Person Responsible _____ Actual completion date _____
Deficiency _____

Action to be taken/Assignment _____

QA follow-up/recurrence audit findings _____
SOP modified? Y or N Target date ___

Auditor _____ CAR closed _____

SPR# 0202

WHITE; ASSIGNEE/RESOLUTION COPY CANARY; CPRTC COPY PINK; CPR TRACKING GOLDENROD; ORIGINATOR COPY

Table 15-1. QC Frequency, Acceptance Criteria, and Corrective Action for Method Blanks

Lab	Method	Frequency	Acceptance Criteria	Corrective Action
GC/MS	624	once/12 hrs	All surrogates within control limits (CL) All target compound lists (TCLs) < 1/2 the detection limit (DL, equal to the reporting limit). All non-TCLs < 25% internal standard (IS) peak height.	Decontaminate lines and trap. Flush trap. Reanalyze until blank meets criteria.
GC/MS	625	minimum 5%	All surrogates within CLs TCL phthalates < 2 x DL No more than five non-TCLs (excluding solvent byproducts) > 25% IS peak height	Halt analysis until problem is identified and corrected. Re-extract entire batch.
GC/MS	8270	minimum 5% aqueous (A) solid (S)	All surrogates within CLs TCL phthalates < 2 x DL Other TCLs < 1/2 DL No more than five non-TCLs > 10% IS peak height No more than three non-TCLs > 25% and < 5 x IS peak height	Halt analysis until problem is identified and corrected. Re-extract entire batch.
GC/MS	8240	once/12 hrs. (A)	All surrogates within CLs TCL common lab solvents < 2 x DL Other TCLs < 1/2 DL No more than five non-TCLs > 10% IS peak height No more than three non-TCLs > 25% and < 5X IS peak height	Decontaminate lines and trap. Flush trap. Reanalyze until blank meets all all criteria.
GC/MS	8240	minimum 5% (S)	All surrogates within CLs TCL common lab solvents < 2 x DL Other TCLs < 1/2 DL No more than five non-TCLs > 10% IS peak height No more than three non-TCLs > 25% and < 5X IS peak height	Halt analysis until problem is identified and corrected. Reprepare entire batch.
GC/MS	CLP 3/90 VOA	once/12 hrs. (A)	All surrogates within CLs TCL common lab solvents < 2 x contract-required quantitation limits (CRQL) Other TCLs < 1/2 CRQL No more than five non-TCLs > 10% IS peak height No more than three non-TCLs > 25% and < 5X IS peak height	Decontaminate lines and trap. Flush trap. Reanalyze until blank meets all criteria.
GC/MS	CLP 3/90 VOA	minimum 5% (S)	All surrogates within CLs TCL common lab solvents < 5 x CRQL Other TCLs < 1/2 CRQL No more than five non-TCLs > 10% IS peak height No more than three non-TCLs > 25% and < 5X IS peak height	Halt analysis until problem is identified and corrected. Re-extract entire batch.

Table 15-1 (CONTINUED). QC Frequency, Acceptance Criteria, and Corrective Action for Method Blanks

Lab	Method	Frequency	Acceptance Criteria	Corrective Action
GC/MS	10/92 SAM VOA	once/12 hrs.	All TCLs < CRQL Surrogates within CLs Non-TCLs < 2.0 ug/L	Decontaminate lines and trap. Flush trap. Reanalyze until blank meets all criteria
GC/MS	CLP 3/90 SV	minimum 5% (A) (S)	All surrogates within CLs except one acid and one base/neutral, but > 10% recovery TCL phthalates < 2 x CRQL Other TCLs < 1/2 CRQL No more than five non-TCLs/non-solvents > 10% IS peak height No more than three non-TCLs > 25% and < 5X IS peak height	Halt analysis until problem is identified and corrected. Re-extract entire batch.
GC/MS	10/92 SAM SV	minimum 5% (A)	All surrogates within CLs TCL ≤ CRQL Non-TCLs < 10 ug/L	Halt analysis until problem is identified and corrected. Re-extract entire batch
GC	601	once/24 hrs.	All surrogates within CLs methylene chloride < 5 ppb all other TCLs < DL	Decontaminate lines and trap. Flush column. Reanalyze until blank meets all criteria. Repeat initial calibration if necessary.
GC	602	once/24 hrs.	Surrogates within CLs All TCLs < DL	Decontaminate lines and trap. Flush column. Reanalyze until blank meets all criteria. Repeat initial calibration if necessary.
GC	608	minimum 5%	Surrogates within CLs All TCLs < DL	Halt analysis until problem is identified and corrected. Re-extract entire batch.
GC	610/8310	minimum 5%	Surrogates within CLs All TCLs < DL	Halt analysis until problem is identified and corrected. Re-extract entire batch.
GC	mod. 8015	minimum 5%	Surrogates within CLs TCLs < DL	Decontaminate lines and trap. Flush trap. Reanalyze until blank meets all criteria. Repeat initial calibration if necessary.

Table 15-1 (CONTINUED). QC Frequency, Acceptance Criteria, and Corrective Action for Method Blanks

Lab	Method	Frequency	Acceptance Criteria	Corrective Action
GC	8010	minimum 10%	Surrogates within CLs Methylene chloride < 5 ppb All other TCLs < DL	Decontaminate lines and trap. Flush column. Reanalyze until blank meets all criteria Repeat initial calibration if necessary.
GC	8020	minimum 10%	Surrogates within CLs All TCLs < DL	Decontaminate lines and trap. Flush column. Reanalyze until blank meets all criteria. Repeat initial calibration if necessary.
GC	8140, 8150 8080	minimum 5%	Surrogates within CLs All TCLs < DL	Halt analysis until problem is identified and corrected. Re-extract entire batch.
GC	CLP 3/90	one/extraction batch; at least one/20 samples	No TCLs > CRQLs Surrogates > 20% recovery	If TCLs > CRQLs, reprepare entire batch.
GC	10/92 SAM Pest/PCBs	one/extraction batch; at least one/20 samples	Surrogate recovery 60-150% No TCLs > CRQL	If blank fails surrogate requirements for 10/92 SAM, re-extract entire batch.
Inorg.	375.4 sulfate 9038	minimum 5%	< reporting limit	Prepare and reanalyze entire batch.
Inorg.	310.2 alkalinity 10-303-31-1-A	minimum 5%	< reporting limit	Prepare and reanalyze entire batch.
Inorg.	10-124-13-1-A hexavalent Cr	minimum 5%	< reporting limit	Prepare and reanalyze entire batch.
Inorg.	325.2 chlorides 9251 10-117-07-1-A	minimum 5%	< reporting limit	Prepare and reanalyze entire batch.
Inorg.	353.2 nitrate 353.2 nitrite	minimum 5%	< reporting limit	Prepare and reanalyze entire batch.
Inorg.	200.7, CLP, 6010 ICP metals	minimum 5%	< CRDL or reporting limit	Prepare and reanalyze entire batch.

Table 15-1 (CONTINUED). QC Frequency, Acceptance Criteria, and Corrective Action for Method Blanks

Lab	Method	Frequency	Acceptance Criteria	Corrective Action
Inorgan.	206.2, CLP, 7060 (As) 270.2, CLP, 7740, (Se) 239.2, CLP, 7421 (Pb) 279.2, CLP, 7841 (Tl) GFAA metals	minimum 5%	< CRDL or reporting limit	Prepare and reanalyze entire batch.
Inorgan.	245.1, CLP, 7470 (A) 245.5, CLP, 7471 (S) Hg by CVAA	minimum 5%	< CRDL or reporting limit	Reprepare and reanalyze entire batch.
Inorgan.	335.2-3, CLP (A) CLP (S) CN	minimum 5%	<CRDL or reporting limit	Prepare and reanalyze entire batch.
Inorgan.	350.1 10-107-06-1-A ammonia	minimum 5%	< reporting limit	Prepare and reanalyze entire batch.
Inorgan.	130.1 10-301-31-1-A hardness	minimum 5%	< reporting limit	Prepare and reanalyze entire batch.
Inorgan.	420.1/2 phenols	minimum 5%	< reporting limit	Prepare and reanalyze entire batch.
Inorgan.	340.2 10-109-12-2-A fluoride	minimum 5%	< reporting limit	Prepare and reanalyze entire batch.
Inorgan.	160.1 filterable residue	minimum 5%	< 10 mg/L of filterable residue	Prepare and reanalyze entire batch.
Inorgan.	160.2 non-filterable residue	minimum 5%	< 4 mg/L of non-filterable residue	Prepare and reanalyze entire batch.
Organic Charact.	418.1 (A) (S) 503E (A) 9071 (S) TPH	minimum 5%	≤ 1.0 ppm (A) ≤ 25 ppm (S)	Prepare and reanalyze entire batch.
Organic Charact.	503B (A) 9071 (S) oil & grease	minimum 10%	≤ 1.0 ppm (A) ≤ 35 ppm (S)	Prepare and reanalyze entire batch.

Table 15-1 (CONTINUED). QC Frequency, Acceptance Criteria, and Corrective Action for Method Blanks

Lab	Method	Frequency	Acceptance Criteria	Corrective Action
Organic Charact.	505 total organic carbon	minimum 10%	≤ 1.0 ppm (A) ≤ 50 ppm (S)	Prepare and reanalyze entire batch.
Organic Charact.	506 total organic	minimum 10%	< 10 mg/kg (S) < 0.01 mg/L (A)	Prepare and reanalyze entire batch.
Radio-chemistry	9310 Gross A/B (A) (S)	minimum 5%	< 1.0 pCi/L/g alpha < 2.0 pCi/L/g beta	Halt analysis until problem is identified and corrected. Recount and assess data for usability.
Radio-chemistry	903.1 radium-226 (A) 9315 total radium (A)	minimum 5%	< 1.0 pCi/L above instrument background	Halt analysis until problem is identified and corrected. Recount and assess data for usability.
Radio-chemistry	HASL300 mod. U-02 (A) isotopic U-234, 238	minimum 5%	< 1.0 pCi/L above instrument background	Halt analysis until problem is identified and corrected. Recount and assess data for usability.

Table 15-2. QC Frequency, Acceptance Criteria, and Corrective Action for Blank Spikes (BS) and Laboratory Control Samples

Lab	Method	Frequency	Acceptance Criteria	Corrective Action
GC/MS	624	minimum 5%	All spike and surrogate recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Repeat BS to verify system control restored.
GC/MS	625	minimum 5%	All spike and surrogate recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Re-extract/reanalyze entire batch to verify system control restored.
GC/MS	8270	minimum 5%	All spike and surrogate recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Re-extract/reanalyze entire batch to verify system control restored.
GC/MS	CLP SV 3/90	minimum 5%	All spike and surrogate recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Re-extract/reanalyze entire batch to verify system control restored.
GC/MS	10/92 SAM SV	one/SDG	All spike and surrogate recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Re-extract/reanalyze entire batch to verify system control restored.
GC/MS	8240, CLP VOA 3/90	minimum 5%	All spike and surrogate recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Repeat BS to verify system control restored.
GC/MS	10/92 SAM VOA	minimum 5%	All spike and surrogate recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Repeat LCS to verify system control restored, then repeat associated samples.
GC	601	minimum 10% (LCS pair)	All spike recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Repeat LCS and MS/MSD to verify system control restored.
GC	602	minimum 10% (LCS pair)	All spike recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Repeat LCS and MS/MSD to verify system control restored.
GC	608	minimum 10% (LCS pair)	All spike recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Re-extract/reanalyze entire batch to verify system control restored.

Table 15-2 (CONTINUED). QC Frequency, Acceptance Criteria, and Corrective Action for Blank Spikes (BS) and Laboratory Control Samples

Lab	Method	Frequency	Acceptance Criteria	Corrective Action
GC	8010	minimum 5%	All spike recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Repeat LCS and MS/MSD to verify system control restored.
GC	8020	minimum 5%	All spike recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Repeat LCS and MS/MSD to verify system control restored.
GC	8080	minimum 5%	All spike recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Re-extract/reanalyze entire batch to verify system control restored.
GC	mod. 8015	minimum 5%	All spike recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Repeat LCS to verify system control restored.
GC	8140	minimum 5%	All spike recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Re-extract/reanalyze entire batch to verify system control restored.
GC	8150	minimum 5%	All spike recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Re-extract/reanalyze entire batch to verify system control restored.
GC	CLP 3/90 Pest/PCBs	minimum 5%	All spike recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Repeat LCS and MS/MSD to verify system control restored.
GC	610/8310	minimum 5%	All spike recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Re-extract/reanalyze entire batch to verify system control restored.
GC	10/92 SAM Pest/PCBs	minimum 5%	All spike recoveries must be within control limits.	Halt analysis until problem is identified and corrected. Repeat LCS to verify system control restored, then repeat associated samples.

Table 15-2 (CONTINUED). QC Frequency, Acceptance Criteria, and Corrective Action for Blank Spikes (BS) and Laboratory Control Samples

Lab	Method	Frequency	Acceptance Criteria	Corrective Action
Inorganics	375.4 9038 sulfate	minimum 5%	±20% true	Prepare and reanalyze entire batch.
Inorganics	325.2 10-117-07-1-A 9251 chloride	minimum 5%	±20% true	Prepare and reanalyze entire batch.
Inorganics	353.2 nitrate/nitrite	minimum 5%	±20% true	Prepare and reanalyze entire batch.
Inorganics	200.7, CLP 3/90, 6010 ICP metals	minimum 5%	±20% true	Prepare and reanalyze entire batch.
Inorganics	310.2 10-303-31-1-A alkalinity	minimum 5%	±20% true	Prepare and reanalyze entire batch.
Inorganics	10-124-13-1-A hexavalent Cr	minimum 5%	±20% true	Prepare and reanalyze entire batch.
Inorganics	350.1 10-107-06-1-A ammonia	minimum 5%	±20% true	Prepare and reanalyze entire batch.
Inorganics	206.2/CLP/ 7060 (As) 270.2/CLP/ 7740 (Se) 239.2/CLP/ 7421 (Pb) 279.2/CLP/ 7841 (Tl) GFAA metals	minimum 5%	±20% true	Prepare and reanalyze entire batch.
Inorganics	245.1, CLP, 7470 (A) 245.5, CLP, 7471 (S) Hg by CVAA	minimum 5%	±20% true	Prepare and reanalyze entire batch.
Inorganics	335.2-3, CLP (A) CLP (S) CN	minimum 5%	±20% true	Prepare and reanalyze entire batch.

Table 15-2 (CONTINUED). QC Frequency, Acceptance Criteria, and Corrective Action for Blank Spikes (BS) and Laboratory Control Samples

Lab	Method	Frequency	Acceptance Criteria	Corrective Action
Inorganics	420.1/.2 phenols	minimum 5%	±20% true	Prepare and reanalyze entire batch.
Inorganics	340.2 10-109-12-2-A fluoride	minimum 5%	±20% true	Prepare and reanalyze entire batch.
Inorganics	130.1 10-301-31-1-A hardness	minimum 5%	±20% true	Prepare and reanalyze entire batch.
Organic Charact.	418.1/503E 9073/9071 TPH	minimum 5%	75-125% recovery	Prepare and reanalyze entire batch.
Organic Charact.	503B 9071 oil & grease	minimum 5%	75-125% recovery	Prepare and reanalyze entire batch.
Organic Charact.	505 505M TOC	minimum 5%	75-125% recovery	Prepare and reanalyze entire batch.
Organic Charact.	506 9020 TOX	minimum 5%	75-125% recovery	Prepare and reanalyze entire batch.
Radiochem.	9310 gross A/B	minimum 5%	±25% of true value	Prepare and reanalyze entire batch.
Radiochem.	903.1 radium-226 (A) 9315 total radium (A)	minimum 5%	±25% of true value	Prepare and reanalyze entire batch.
Radiochem.	U-02 isotopic uranium-234,238	minimum 5%	±25% of true value	Prepare and reanalyze entire batch.

Table 15-3. QC Frequency, Acceptance Criteria, and Corrective Action for Matrix Spikes (MS) and MS/MSD Pair

Lab	Method	Frequency	Acceptance Criteria	Corrective Action
GC/MS	624	minimum 5% full MS/MSD pair or single MS	All spike recoveries must be within control limits.	Analyze BS. If BS is acceptable, report MS/MSD and BS with data qualifier noting sample matrix interference.
GC/MS	625	minimum 5% full MS/MSD pair or single MS	All spike recoveries must be within control limits.	Analyze BS. If BS is acceptable, report MS/MSD and LCS with data qualifier noting sample matrix interference.
GC/MS	8270	minimum 5% MS/MSD pair	Majority of spike recoveries and RPDs must be within control limits.	Analyze BS. If BS is acceptable, report MS/MSD and LCS with data qualifier noting sample matrix interference.
GC/MS	8240	minimum 5% MS/MSD pair	Majority of spike recoveries and RPDs must be within control limits.	Analyze BS. If BS is acceptable, report MS/MSD and LCS with data qualifier noting sample matrix interference.
GC/MS	CLP 3/90 VOA	minimum 5% MS/MSD pair	Majority of spike recoveries and RPDs must be within control limits.	Analyze BS. If BS is acceptable, report MS/MSD and LCS with data qualifier noting sample matrix interference.
GC/MS	CLP 3/90 SV	minimum 5% MS/MSD pair	Majority of spike recoveries and RPDs must be within control limits.	Analyze BS. If BS is acceptable, report MS/MSD and LCS with data qualifier noting sample matrix interference.
GC	601	minimum 10%	Majority of recoveries and RPDs must be within control limits.	Analyze BS. If BS is acceptable, report MS/MSD and LCS with data qualifier noting sample matrix interference.
GC	602	minimum 10%	Majority of recoveries and RPDs must be within control limits.	Analyze BS. If BS is acceptable, report MS/MSD and LCS with data qualifier noting sample matrix interference.

Table 15-3 (CONTINUED). QC Frequency, Acceptance Criteria, and Corrective Action for Matrix Spikes (MS) and MS/MSD Pair

Lab	Method	Frequency	Acceptance Criteria	Corrective Action
GC	608	minimum 10%	Majority of recoveries and RPDs must be within control limits.	Analyze BS. If BS is acceptable, report MS/MSD and LCS with data qualifier noting sample matrix interference.
GC	8010	minimum 10%	Majority of recoveries and RPDs must be within control limits.	Analyze BS. If BS is acceptable, report MS/MSD and LCS with data qualifier noting sample matrix interference.
GC	8020	minimum 10%	Majority of recoveries and RPDs must be within control limits.	Analyze BS. If BS is acceptable, report MS/MSD and LCS with data qualifier noting sample matrix
GC	8080	minimum 10%	Majority of recoveries and RPDs must be within control limits.	Analyze BS. If BS is acceptable, report MS/MSD and LCS with data qualifier noting sample matrix
GC	8140	minimum 10%	Majority of recoveries and RPDs must be within control limits.	Analyze BS. If BS is acceptable, report MS/MSD and LCS with data qualifier noting sample matrix
GC	8150	minimum 10%	Majority of recoveries and RPDse must be within control limits.	Analyze BS. If BS is acceptable, report MS/MSD and LCS with data qualifier noting sample matrix
GC	CLP 3/90 Pest/PCBs	minimum 5%	Majority of recoveries and RPDs must be within control limits.	Analyze BS. If BS is acceptable, report MS/MSD and LCS with data qualifier noting sample matrix
GC	610/8310	minimum 10%	Majority of recoveries and RPDs must be within control limits.	Analyze BS. If BS is acceptable, report MS/MSD and LCS with data qualifier noting sample matrix
Inorg.	200.7, CLP, 6010 ICP metals	minimum 5% (sample spike)	75-125% recovery* (See footnote at end of table.)	Flag data with "N".

Table 15-3 (CONTINUED). QC Frequency, Acceptance Criteria, and Corrective Action for Matrix Spikes (MS) and MS/MSD Pair

Lab	Method	Frequency	Acceptance Criteria	Corrective Action
Inorganics	206.2/CLP/ 7060 (As) 270.2/CLP/ 7740 (Se) 239.2/CLP/ 7421 (Pb) 279.2/CLP/ 7841 (Tl) GFAA metals	minimum 5% (sample spike)	75-125% recovery* (See footnote at end of table.)	Flag data with "N".
Inorganics	245.1 CLP, 7470 (A) 245.5, CLP, 7471 (S) Hg by CVAA	minimum 5% (sample spike)	75-125% recovery* (See footnote at end of table)	Flag data with "N".
Inorganics	335.2-.3 CLP, 7470 (A) CLP (S) CN	minimum 5% (sample spike)	75-125% recovery* (See footnote at end of table)	Flag data with "N".
Inorganics	420.1/.2 phenols	minimum 5%	75-125% recovery** (See footnote at end of table)	QA Notice is required.
Inorganics	340.2 10-109-12-2-A fluoride	minimum 5%	75-125% recovery*** /RPD $\leq$ 20 (See footnote at end of table)	QA Notice is required.
Inorganics	375.4 9038 sulfate	minimum 5%	75-125% recovery*** /RPD $\leq$ 20 (See footnote at end of table)	QA Notice is required.
Inorganics	325.2 10-117-07-1-B 9251, chloride	minimum 5%	75-125% recovery*** /RPD $\leq$ 20 (See footnote at end of table)	QA Notice is required.
Inorganics	353.2 nitrate/nitrite	minimum 5%	75-125% recovery*** /RPD $\leq$ 20 (See footnote at end of table)	QA Notice is required.
Inorganics	310.2 10-303-31-1-A alkalinity	minimum 5%	75-125% recovery*** /RPD $\leq$ 20 (See footnote at end of table)	QA Notice is required.

Table 15-3 (CONTINUED). QC Frequency, Acceptance Criteria, and Corrective Action for Matrix Spikes (MS) and MS/MSD Pair

Lab	Method	Frequency	Acceptance Criteria	Corrective Action
Inorganics	10-124-13-1-A hexavalent Cr	minimum 5%	75-125% recovery*** /RPD ≤ 20 (See footnote at end of table)	QA Notice is required.
Inorganics	350.1 10-107-06-1-A ammonia	minimum 5%	75-125% recovery*** /RPD ≤ 20 (See footnote at end of table)	QA Notice is required.
Inorganics	130.1 10-301-31-1-A hardness	minimum 5%	75-125% recovery*** /RPD ≤ 20 (See footnote at end of table)	QA Notice is required.
Organic Charact.	418.1/503E 9071 TPH	minimum 10%	75-125% recovery /RPD ≤ 25	Analyze LCS or reprepare and reanalyze MS/MSD.
Organic Charact.	503B 9071 oil & grease	minimum 10%	75-125% recovery /RPD ≤ 25	Analyze LCS or reprepare and reanalyze MS/MSD.
Organic Charact.	505/505M TOC	minimum 10%	75-125% recovery /RPD ≤ 25	Analyze LCS or reprepare and reanalyze MS/MSD.
Organic Charact.	506 9020 TOX	minimum 5%	60-120% recovery /RPD ≤ 25	Analyze LCS or reprepare and reanalyze MS/MSD.

Footnotes

* A majority of percent recoveries (%R) must fall within the acceptance range, or repreparation of the entire sample batch must occur unless the limits are advisory and holding time has expired. If an individual recovery is not within acceptance criteria, the data for all samples associated with the spiked sample and determined by the same analytical method must be flagged with an "N" on Forms I and V. If the sample concentration exceeds the spike concentration by a factor of four or more, the data is reported unflagged, even if the spike recovery is outside the acceptance range.

** Exceptions will be defined in a QA Notice.

*** Based on standard aqueous sample matrix or documented methods for solid preparations. If recovery is not within control limits and sample result does not exceed 4 x the amount of spike added, matrix interference is suspected. Verify by spiking another aliquot of the sample. If control limits are still not met, a QA Notice is required.

Table 15-4. QC Frequency, Acceptance Criteria, and Corrective Action for Duplicates

Lab	Method	Frequency	Acceptance Criteria	Corrective Action
Inorganics	200.7 CLP, 6010 ICP metals	minimum 5%	20% RPD* (See footnotes at the end of the table.)	Flag data outside acceptance criteria with an "*". **
Inorganics	206.2/CLP/ 7060 (As) 270.2/CLP/ 7740 (Se) 239.2/CLP/ 7421 (Pb) 279.2/CLP/ 7841 (Tl) GFAA metals	minimum 5%	20% RPD* (See footnotes at the end of the table.)	Flag data outside acceptance criteria with an "*".
Inorganics	245.1, CLP/ 7470 (A) 245.5, CLP/ 7471 (S) Hg by CVAA	minimum 5%	20% RPD* (See footnotes at the end of the table.)	Flag data outside acceptance criteria with an "*".
Inorganics	325.2-.3 CLP, 6010 CN (A) CLP (S) CN	minimum 5%	20% RPD* (See footnotes at the end of the table.)	Flag data outside acceptance criteria with an "*".
Inorganics	160.1 filterable residue	minimum 5%	20% RPD** (See footnotes at the end of the table.)	QA Notice required.
Inorganics	160.2 non-filterable residue	minimum 5%	20% RPD** (See footnotes at the end of the table.)	QA Notice required.
Inorganics	420.1-.2 phenols	minimum 5%	20% RPD** (See footnotes at the end of the table.)	QA Notice required.

Footnotes

* The ±20% RPD is used when the sample and duplicate results are greater than or equal to five times the CRDL or reporting limit. A control limit of ± the CRDL/reporting limit is used if either the samples or duplicate value is less than five times the CRDL/reporting limit. If both the duplicate and sample values are less than the IDL, then the RPD is not calculated and no acceptance criteria can be applied. If an individual RPD is not within acceptance criteria, the data for all samples associated with the duplicate must be flagged with an asterisk (*) on Forms I and VI.

** Exceptions will be noted in a QA Notice.

Table 15-4. QC Frequency, Acceptance Criteria, and Corrective Action for Duplicates

Lab	Method	Frequency	Acceptance Criteria	Corrective Action
Radiochemist	9310/703 gross A/B	minimum 10%	±25% RPD	Recount and assess data for usability.
Radiochemist	903.1 radium-226 (A) 9315 total radium (A)	minimum 10%	±25% RPD If samples <5 x the blank, duplicate must be ±5 pCi/L	Recount and assess data for usability.
Radiochemist	U-02 isotopic U-234,238	minimum 10%	±25% RPD	Recount and assess data for usability. If samples <5 X the blank, duplicate must be ±5 pCi/L.

16.0 QA Reports to Management

Each QA department staff member produces a monthly report summarizing QA activities during the previous month. The QA manager submits these reports to the Vice President General Manager, who includes them in his cumulative report to the Chief Executive Officer (CEO) and the senior executive staff.

All laboratory managers receive a portion of the QA report that contains a statistical analysis of production and repeat rates. Laboratory managers use the report to numerically assess total monthly production, frequency of repeated analyses, and causes of unacceptable conditions or performance. QA presents these data in a series of Pareto charts, line graphs, and bar graphs. QA obtains this information from the LIMS based on the frequency of analytical condition codes applied to samples.

Condition codes are two-letter codes used to indicate the status of each analysis in the LIMS. This system of codes is used to monitor and track analytical performance. Data reviewers or analysts assign appropriate codes to each analysis. For example, if surrogate recovery in a GC/MS semivolatile analysis falls below the lower control limit, the data reviewer assigns the code *SL* (surrogate recovery low). The data reviewer then documents this by recording the code on the appropriate portion of the analytical worksheet. The code is then entered into the schedule detail record (*queue*) in the LIMS so that the sample can be scheduled for reextraction and reanalysis. This record is useful to identify when additional feedback, performance testing, or training may be needed. More than 50 condition codes have been defined for this system.

Various computer programs are used to sort and to assess the condition code and schedule detail record by sample matrix, sample type, analytical method, and procedure (SOP). QA staff note in their monthly reports any procedural changes necessary as a result of applying condition codes.

The monthly report may include a summary of quality problems observed during the period, and any corrective actions taken to prevent recurrences of the problems. The report stresses proactive measures QA takes to improve total quality and to ensure compliance with QA program requirements.

The supervisor of the Technical Communications department, who controls all SOPs, laboratory logbooks, and the QA Plan, and administers all laboratory certifications, also submits a monthly report to the VPGM. This report summarizes document revision and distribution as well as the status of all certifications of the laboratory by external agencies.

The QA department report includes:

- information on QA's role in resolving quality issues with clients or other external agencies
- notification of any QA organizational changes
- information on training and safety issues that were not covered in audit reports during the period
- performance summaries of subcontractor laboratories
- summaries of self-inspection activities
- feedback for acceptable performance on interlaboratory or intralaboratory tests and audits
- summary of customer problem resolution activities, including outstanding and closed CPRRs
- information on periodic QA assessments of measurement data accuracy, precision, and method detection limits
- summaries of external audits conducted

The QA manager summarizes all routine system auditing activities conducted by the QA department during each quarter and reports this to the VPGM. The VPGM uses information about the outcome of QA activities to assess the effectiveness of quality management systems and to identify problems that hinder successfully meeting quality program objectives such as adequacy of resources and personnel, or effectiveness of management controls. Management responses to corrective action should be prompt and are evaluated for effectiveness.

17.0 Facilities and Safety

CompuChem Environmental Corporation is located in two adjoining buildings in Research Triangle Park (RTP), North Carolina, which is 15 miles west of Raleigh. One of the largest GC/MS laboratories in the world is housed at our RTP facility. The laboratory facility has over 38,000 square feet of office and laboratory space. Over 29,000 square feet of this space contains the laboratory areas (Figure 17-1), while the remainder contains support areas.

We use state-of-the-art instruments and data processing equipment for organic, inorganic, and mixed waste analyses. One Hewlett Packard (HP)-3000 series 937 is dedicated to scheduling and tracking analyses through the laboratories. An additional HP-3000 series 70 computer is used for the accounting systems. Other computing resources in our laboratory include a PDP-11, three HP-1000s, a NOVA 4C or 4X system built into each of our GC/MS instruments, and more than 100 microcomputers. The LIMS is accessed by laboratory, marketing, systems management, and accounting personnel via more than 130 computer terminals located throughout the facility. A comprehensive instrument inventory list is available as Supplement C to this QA Plan. For ordering information, see Section 3.0.

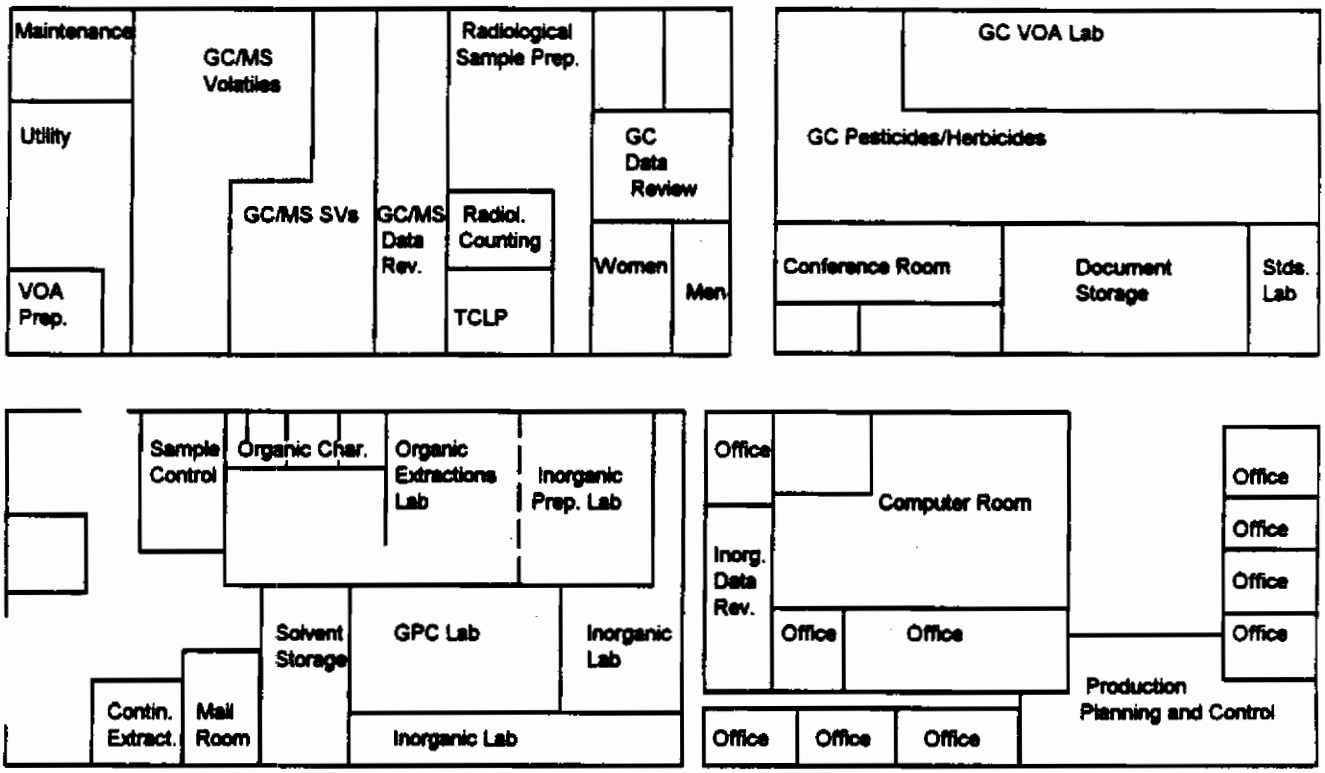
The environmental controls for heating and cooling start and stop automatically to maintain temperature control. All areas in which temperature is critical, such as walk-in and reach-in refrigerators, freezers, and computer rooms, are monitored 24 hours per day by an off-site security monitoring firm. This ensures that proper temperatures are maintained. Temperatures are documented in logbooks twice daily for these units to provide a historical record. The fire and burglar alarms are also monitored by this firm, which will contact the appropriate personnel at state and local agencies in the event of an emergency.

A security guard is on site seven days a week, 24 hours a day. Building access is restricted to authorized CompuChem personnel. Rusco card entry readers are in place at all building entrances and exits and at entrances to the laboratory building. All critical laboratory areas are continuously supported by an uninterrupted power supply (UPS) system in the event of a power interruption. Only employees requiring entry to perform their job functions are allowed access to restricted areas. Each employee must have identification on them while on the premises. This ID must include the employee's picture and personal identification number.

17.1 Laboratory Areas

The laboratory areas at CompuChem include the Sample Control department, the walk-in refrigerator systems, the Organic and Inorganic Sample Preparation Laboratory, the solvent storage area, the GC/MS Laboratory, the TCLP Laboratory, the ZHE Laboratory, the GC Laboratory, the Radiological Laboratory, the Standards Laboratory, the Inorganic Instrument Laboratory, the Organic Characterization Laboratory, the GPC/Florisil Laboratory, separate continuous liquid-liquid extraction room, the radiological sample preparation and counting rooms, and the extract storage area. An instrument maintenance area is in the expansion building adjacent to the single-story laboratory building.

Figure 17-1 **CompuChem Laboratories Floor Plan (Laboratory Building)**



Scale: None

Table 17-1. Facilities Space Allocation

Department	Square Footage
Laboratory Management	348
Sample Control	2479
Production Planning and Control	462
Report Preparation/Document Control/Copy Room	4576
Technical Review	895
Glassware Preparation	742
Solvent Storage	536
GPC Room	446
Continuous Extraction	420
Leachate Preparation	273
Quality Assurance	605
Technical Communications	250
Instrumentation	1632
Radiological Laboratory	1451
GC Laboratory	2281
GC/MS VOA Laboratory	2091
GC/MS Semivolatile Laboratory	2768
Inorganics Laboratory	4208
Laboratory Automation	560
Warehouse	68
Facilities	474
Computer Operations	1540
Sales	1024
Customer Service/Project Management	1762
General Management	1107
Human Resources	509
Finance	1071

Sample Control
The Sample Control Department is adjacent to the laboratories. Samples arriving are identified and logged into the LIMS for scheduling and tracking.

Walk-in Refrigerator Systems
Two separate refrigeration systems are used for sample storage, one of which is used exclusively for storing samples scheduled for volatile organic analyses. The units are two independent refrigeration systems that are kept at a temperature of 4°C ± 2°C, and that are equipped with activated carbon air filtering systems. Both refrigerators are locked, with access restricted to sample custodians, and the temperature-activated alarm systems are monitored by a private security service. In the event of unauthorized entry or temperature fluctuations, appropriate parties are notified. If power is interrupted a UPS, in conjunction with a gas-powered generator, maintains the appropriate temperatures until the electricity is restored by Duke Power Company. The laboratory has at its disposal a refrigerated tractor trailer in the event of catastrophic power failure.

Sample Preparation Laboratories
These laboratories include an area for organic sample preparation and an area for inorganic sample preparation. Each area is equipped with ventilation hoods. A single-pass air handling system is in place in these laboratories acting, in effect, as a walk-in hood. Fresh air is introduced into the area through the ceiling and is forced out near the floor. The system exchanges the laboratories' air every two minutes. Continuous liquid-liquid extractions are performed in a separate laboratory area which has the capacity for up to 80 continuous extractors.

Toxicity Characteristic Leaching Procedure (TCLP) Preparation Laboratory
This area is equipped to perform TCLP on aqueous and solid samples for semivolatile GC/MS, pesticide and herbicide GC, and metals analysis. The laboratory is equipped with six rotators that can hold eight samples each, four Millipore waste filtration devices for filtering leachates, an exhaust hood, and a deionized water source. The room temperature is monitored when leachates are being prepared. In addition, the pH meter is calibrated using three certified buffers (2.0, 4.0, and 10.0) to bracket the buffers used in the TCLP.

Zero Headspace Extraction (ZHE) and Solid VOA Preparation Laboratory
This area is equipped to perform the ZHE on samples for volatile GC/MS analysis. The laboratory is equipped with three rotators each equipped to hold eight samples, 20 Millipore ZHE devices, and an exhaust hood. The room temperature is monitored when leachates are being prepared.

Solvent Storage Area

The solvent storage area is adjacent to the sample preparation laboratories, and is secured at all times. Its safety features include reinforced concrete walls, alarm systems, a Halon fire suppression system, and a roof designed to implode to relieve pressure in the event of an accident.

GC/MS Laboratory

CompuChem's 22 GC/MS instrument systems (15 organic water analyzers [OWAs] and seven INCOS-500s) are strategically placed on a raised computer floor. This allows the gas, water, cooling, and exhaust systems required to support each instrument to enter and leave the room independently beneath the floor. Equipment is arranged in working clusters for maximum efficiency. In this way, specific instruments can be used for specific types of analyses. All OWA instruments used for semivolatile analyses have been upgraded with Varian 3400 GCs, CTC autosamplers, operating systems, and heated sources, as appropriate for the instrument application.

The volatile laboratory and the semivolatile laboratory are separated by floor-to-ceiling glass walls. To prevent cross-contamination, instruments used for volatile analyses are never used for semivolatile analyses. The volatile laboratory's intake air is carbon-filtered. As a further precaution against contamination, the volatile laboratory has positive pressure, which causes air to go out of but not into the volatile laboratory from other areas in the building. Moreover, each cluster of instruments is staffed by analysts cross-trained to perform each procedure. This staffing system permits intimate daily interactions between the operator, the instruments, and applied methodologies. All other instruments are dedicated in a similar way.

These GC/MS systems are all connected to the CompuChem computer network to assure accurate data transmission and archiving of analytical results. This networking feature reduces data processing time and protects data integrity.

GC Laboratory

The laboratory's 29 gas chromatographs are equipped with autosamplers or purge and trap devices (Tekmar LSC-2) and are interfaced with HP 1000 laboratory computers for data processing. Many of the GCs are equipped with dual detectors. A variety of detectors are attached to the GCs, including flame ionization, flame photometric, electron capture, thermionic-specific (also called nitrogen-phosphorus or alkaline-flame ionization) electrocoulometric (Hall) detectors. The laboratory is also equipped with three high performance liquid chromatographs (HPLCs) with autosamplers. These instruments have fluorescence and/or UV detectors.

Radiological Laboratory

This laboratory is separated completely from all other laboratories, and is divided into two functional areas, a radiochemistry laboratory and a counting room. The laboratory is equipped with four eight-foot ventilation hoods, 64 square feet of bench space, storage cabinets, and a sink. The hoods are equipped with HEPA filters. The labora tory has a constant supply of deionized water.

In the counting room are several instruments that detect low level radionuclides. The gamma spectroscopy system consists of an HPGe detector interfaced with an HP Vectra personal computer. The laboratory also includes a Tennelec low-background alpha/beta counter, a Packard 2500TR liquid scintillation analyzer, two Lucas cell counters for radium detection, an NMC PC-55 alpha/beta counter, and 12 alpha spectrometer modules.

Standards Laboratory
This area is separated completely from all other laboratories. Refrigeration, glove box, and hood units are located in this area. A five-place Mettler analytical balance is used for weighing neat materials.

Inorganics Laboratory
The Inorganics Laboratory is separated completely from all other laboratories and is equipped with two simultaneous ICP units, two Technicon autoanalyzers with multiple modules, eight atomic absorption units, one UV-visible spectrophotometer, one Leeman autoanalyzer, and one Lachat autoanalyzer. Several other analytical instruments that perform classical analyses are also located in the laboratory. Hood systems for instrument exhaust are also an integral part of the Inorganics Laboratory. The analytical instruments enable CompuChem to meet even the most stringent and varied inorganics testing requirements.

Organic Characterization
This laboratory prepares and analyzes environmental samples for TPH and oil and grease using an FT-IR (Fourier Transform-Infrared spectrophotometer), TOC using a Dohrmann TOC analyzer, and TOX using a Dohrmann TOX analyzer. Using these instruments, CompuChem is able to screen highly contaminated samples.

Extract Storage
Sample extracts are stored in custom-designed reach-in refrigeration units. These refrigeration units are accessed only by sample custodians; access requires a key. Temperature readings are documented twice daily in standardized logbooks. The refrigeration units are equipped with a temperature-activated alarm system that is monitored by a private security service. In the event of a temperature excursion, appropriate parties are notified.

17.2 Hazardous and Mixed Waste Disposal

CompuChem Environmental Corporation is a large quantity waste generator and is subject to annual inspections. We must comply with all RCRA and State of North Carolina Hazardous Waste Section requirements identified in 40 CFR Part 261.

Mixed and radioactive wastes are handled and disposed of in accordance with the requirements and regulations defined in our radioactive materials license issued by the State of North Carolina. During the process of analytical testing, the laboratory generates hazardous wastes as defined by RCRA. These types of wastes fall primarily under waste stream codes F001, F002, F003, and F005, and are identified in 40 CFR Part 261. Radioactive waste and mixed waste are also generated during the course of radiochemical analyses.

Mixed waste means waste that is both hazardous and radioactive. These wastes are stored at the facility until they are picked up for disposal. All storage drums must be labeled properly, tightly sealed, and overpacked if structural integrity is suspect. Waste handling and disposal procedures conducted on site are carried out in compliance with CompuChem's waste disposal SOP and in compliance with RCRA regulations. All recycling and arrangements for final disposition (i.e., transport to burial or incineration) are handled by a licensed hazardous waste contractor or mixed/ radioactive waste contractor. Unused raw samples that underwent radiological or mixed waste analyses are shipped back to the client as required by CompuChem's radioactive materials license issued by the State of North Carolina. After analysis and following a three month holding period, all residual solid and aqueous samples are disposed of as hazardous waste. CompuChem makes aggressive efforts to minimize waste, and makes every effort to recycle those materials and chemicals that are well suited for this purpose. Table 17-2 describes CompuChem's general waste disposal procedures.

Table 17-2. General Waste Disposal Procedures

Type	Analyses	Storage Conditions	Disposal Procedures
halogenated solvents methylene chloride	pesticides/herbicides/ BNA	55-gallon drums	recycled
Freon	oil and grease/TPH	55-gallon drums	recycled
mixed solvents (flammable & non-halogenated)	VOA standards/herb-icides/pesticides	55-gallon drums	disposed
neat & diluted standards	all	original glass or plastic bottles are lab packed	disposed
heavy metals	metals/chloride	plastic containers	disposed
acid solutions	metals/general inorganics extractables	add to neutralizing chambers	sanitary sewer
all samples	all analyses	original container in Sample Control	disposed
radioactive and mixed waste unused raw samples	radiologicals	original containers in Sample Control	returned to client for disposal

17.3 Health and Safety

CompuChem is concerned about the health and safety of all employees and, therefore, provides a smoke-free workplace. CompuChem has the proper facilities, equipment, and maintenance for a safe and healthy working environment. All laboratory personnel regularly undergo physical examinations. CompuChem's safety officer coordinates many safety-related actvities, including internal and external safety committee inspections. The safety officer also arranges safety inspections by the county fire chief. Fire drills are performed quarterly.

Standard operating procedures that relate to laboratory safety are available throughout the facility and, in the event of an emergency, provide instructions. Evacuation routes are posted within each laboratory and throughout the building. When laboratory spills or accidents occur, laboratory personnel must follow strict

cleanup or first aid procedures, and the area manager must complete a Spill Report or Accident Report and submit it to the company chemical hygiene officer (CHO).

The CHO is responsible for conducting health and safety surveys to identify potential health hazards and collect background information for assessment of these hazards. The CHO documents obvious signs of exposure by spending time in each lab, noting the following:

- airborne dust, smoke, or mist
- accumulations of dust, liquid, or solid residue on the floor, instruments, or work benches
- odors from solvent vapors or gases
- burning or throat/nose irritation
- presence of procedures for responding to emergencies, such as chemical spills, leaks, explosions, and fire

The CHO is also responsible for monitoring quarterly inspections and conducting random inspections to endure that labs meet requirements necessary to mantain a safe working environment.

The CHO also specifies chemicals exposure evaluation strategies to ensure that exposure of personnel to hazardous materials are within acceptable limits. To do this, the CHO compiles a list of hazardous chemicals used at CompuChem and their associated permissible exposure limits (PELs) as defined in 29 CFR Part 1910, subpart Z, of the *Federal Register*. The CHO measures appropriate PELs in each area, using page 3316 of the *Federal Register* (Volume 55, No. 21, January 31, 1990) and page 4 and Appendix B of *Prudent Practices for Handling Hazardous Chemicals in Laboratories* as guides.

If potential risks are identified, the CHO and the lab managers should structure a sampling plan to ensure that exposures are within PELs. The nature of this sampling plan depends on the initial PEL measurements. Following are some monitoring methods that the CHO may use:

- personal sampling
- area air monitoring
- wipe monitoring
- biological monitoring

Through Human Resources, the CHO also coordinates training sessions and information exchange to ensure that employees receive training in safe work practices and emergency response and are made aware of hazards in their areas. Training topics include:

- right to know
- chemical spills
- first aid/CPR
- other appropriate categories
- interpretation of MSDSs
- safe work practices
- waste handling
- use of emergency equipment
- handling radiolabeled materials

17.4 Radiation Safety Officer

The RSO is responsible for overseeing the safety aspects of the radiological operations in the laboratory. The RSO is responsible for:

- periodic certification of the radiation monitoring devices in the sample receiving area and the radiological laboratory
- handling film badge distribution and associated paperwork
- granting final approval of requests to purchase radioactive materials to ensure that quantities do not exceed the limitations of CompuChem's license
- making sure that radioactive materials and wastes received or generated by CompuChem are disposed of properly
- performing monthly swipe tests of the radiological laboratory and related areas of CompuChem in which radioactive materials are used
- performing quarterly inspections of the Radiological Laboratory and related laboratories and issuing a report that lists results, conditions, improvements, etc.
- maintaining records and documentation related to all of the above items

17.5 Radiation Safety Practices

The Radiological Laboratory staff performs several procedures to ensure the safety of personnel within the Radiological Laboratory and personnel in other areas of the laboratory building. When any portion of a prepared standard, blank spike, or tracer is used, the information is recorded in the radiological materials traceability logbook. Whenever a prepared or purchased radiochemical material is disposed of, the disposal is recorded in either the acidic or the basic radiochemical liquid waste disposal logbook. A radiochemist performs a daily survey to monitor background radiation in the sample preparation area and the counting room (areas within the Radiological Laboratory). Any trends are noted. The Radiation Safety Officer (RSO) is notified of any background radiation in the sample preparation area and the counting room (areas within the Radiological Laboratory) so that decontamination procedures can be implemented. As our license requires, we have frisking stations available for personnel leaving the Radiological Laboratory area.

17.6 Chemical Hygiene Plan

CompuChem has developed a written description of our Chemical Hygiene Plan (CHP), which complies with the standards of the Occupational Safety and Health Administration (OSHA) set forth in 29 CFR Part 1910.1450 (January 31, 1990). This CHP incorporates CompuChem's contingency plan.

17.7 Radiological Laboratory Safety Manual

A Radiological Safety Manual is available to all employees and was written for personnel involved in processing radiological samples. The manual is provided during employee orientation and training, and serves as a basic reference for general information. The manual complies with North Carolina regulations under Title 10 CFR.

17.8 Sources

National Research Council. (1990). *Prudent practices for handling hazardous chemicals in laboratories*. National Academy Press, Washington, DC.

Federal Register (January 31, 1990). 29 Code of Federal Regulations Part 1910, II. 55:21.

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Stricoff, R. Scott, and Walters, Douglas B. (1990). *Laboratory health and safety handbook*. John Wiley and Sons, Inc. New York.

U.S. Department of Labor. (1986). *Occupational safety and health standards for general industry*. Promulgated by OSHA based on 20 CFR 1910, Subpart Z, 1910.1200, "Hazard Communication."

18.0 Procurement Control

This section addresses material procurement and control, material quality inspection, and reagent storage.

18.1 Material Procurement and Control

The two prime objectives of CompuChem's Accounting department are to maintain sufficient supplies of all required items as needed, and to encourage all forms of competition to aggressively seek the best total value in a combination of supply, price, required quality, and service. Department and laboratory managers have primary responsibility for maintaining adequate inventory of supplies and ensuring that all supplies/equipment meet or exceed quality requirements. Managers work through the Accounting department to meet these objectives. CompuChem uses competitive inquiries or requests for bids, along with appropriate negotiation, to provide equal opportunities for potential and current suppliers to earn CompuChem's business and to allow the laboratory to seek the best overall value. Long-term considerations include reliability, price, required quality, and service. Suppliers must maintain the confidentiality of competitively sensitive information that is obtained from the Accounting department or other CompuChem personnel. Prices and related information, whether accepted or not, will not be disclosed.

Each year, various vendors will supply the laboratory with solvent/chemical samples during the bidding process. The laboratory evaluates each vendor's sample, as described in the next section, before the bid is considered by the Accounting department. If solvent/chemical quality is equivalent, then price and service are considered. Prices are kept low because of the highly competitive market and the high volume used by the laboratory.

18.2 Material Quality Inspection

Managers interact with the department when purchasing supplies/equipment that could potentially affect data quality, and therefore results of sample analyses, before use in production. The manager of QA or designated QA staff member determines the appropriate test procedures and evaluates the resulting test data. A similar validation process is used in testing new instrumentation. When variability is exhibited in the quality of vendor-supplied materials or services, the laboratory department manager is responsible for working with the Accounting department to find a suitable alternative source.

Information on new chemicals must be supplied to the chemical hygiene officer and safety officer and the waste management office before purchase. Items and services have been identified that are known to affect quality. Control over these is described as follows.

Control of Analytical Standards

High purity standards are purchased through reputable vendors with certificates of traceability to NIST when possible. Certificates are maintained on file for documentation purposes or may be included in the data deliverables. New working level standard lots are evaluated and approved before they are used in production. Organic standards used in non-CLP methods are prepared internally in the organic standards laboratory. The standards chemist is responsible for maintaining adequate inventories and initiating standard purity testing for each preparation lot. QC reference materials traceable to NIST, as available, are supplied to the laboratories by the QA department in conjunction with the single blind PE program. Purchase and inventory of these certified materials are controlled by QA.

Reagent Control

Solvents are purchased in bulk lot quantities. Before purchasing solvents, a bottle from the lot is tested in the laboratories. Solvent lot approval must be documented by the laboratory manager and the QA department before distribution to the supplier warehouse. SOPs are written that define criteria for solvent lot acceptance. An inventory and supply control are maintained by the warehouse. The laboratory continually monitors the integrity of these materials by routinely analyzing QC method blanks with each batch of samples. Chemicals are routinely monitored and inventoried by the appropriate laboratory manager and replenished as needed, allowing adequate time for the order processing, shipment, and quality verification steps.

Radiological Inventory Control

Radiological standards are inventoried at least semiannually to ensure compliance to our licensing limits. All radioactive materials are maintained under inventory control and total activities computed to ensure that allowed quantities of radionuclide activity are not exceeded. Documentation of the inventories are available for audit purposes. Accountability of sample quantities is accomplished by means of a tracking logbook with procedures described in a written SOP. These tracking procedures are performed on an ongoing basis.

Glassware Lot Control

Glassware for use in SampleSaver shipping containers is purchased certified clean. The results from the test analysis of each lot is reviewed by the QA department before purchase. Once approved, the lot is shipped to the vendor warehouse for distribution to the laboratory on an as needed basis. Certificates of analysis are retained in the QA department and made available to our clients upon request. The vendor approval process involves an initial on-site evaluation of the supplier operation and includes the supplier storage warehouse.

Control of Subcontracted Analytical Services

CompuChem performs a wide array of analytical methodologies, and on an exception basis, the laboratory must locate a suitable alternate laboratory to perform methods not performed in house. The use of a qualified laboratory for subcontracted analytical services is agreed to by CompuChem's client before the analyses take place. The QA department oversees subcontract laboratories through a program of inspection and approval. An evaluation of the laboratory includes an on-site audit, review of standard operating procedures, QAPP, and statement of qualifications, and recent PE study scores with laboratory responses to any deviation from CompuChem's quality requirements. Formal approval is granted and renewed annually. The approved supplier list is distributed to in-house personnel who are responsible for procuring subcontracted services.

Control of Computer Hardware and Software

Procedures for computer systems development and validation include an initial survey, feasibility study, analysis and general design, detail design, system development and programming, implementation and production. Each phase is controlled by the Systems and Laboratory Automation department, and are supported by written standard operating procedures. Software validation procedures are performed by means of in-use tests on data reduction spreadsheets used in each laboratory. Hand-made calculations are compared to electronically generated calculations. Acceptable performance of the computer program in the operating system is verified. Validated software are assigned version numbers and version control is maintained by the appropriate laboratory. Results of these test procedures are documented.

Computer hardware maintenance contracts are provided by the original supplier or through third party agreement. Individual users define requirements for the items needed. Internal systems support staff assist users in this process as needed. In service inspection is provided internally by trained hardware and software experts, as needed. Purchase orders are completed and equipment approved by senior staff before the order is placed through the Purchasing department.

Reagent Storage

Table 18-1 presents information about conditions under which reagents are stored. The sample control department stamps each case of reagent with the date received. The reagent supplier ships cases from an approved lot to the laboratory as needed until the lot is depleted. Unused cases are stored in a designated solvent storage room. Reagents within individual laboratories are stored in appropriate cabinets or under hoods. All solutions and reagents are labeled with the date opened/prepared and the opener's/preparer's initials unless the solution or reagent is consumed within one shift.

Table 18-1. Reagent Storage Requirements

Reagent	Storage	Location	Conditions
halogenated solvents	vented storage cabinets	Organic Characterization Lab, Organic Extractions Lab Electronics Shop GC/MS Lab GC Lab Standards Lab	air conditioning
nonhalogenated solvents	vented storage cabinets	Organic Extractions Lab, Inorganic Extractions Lab Electronics Shop GC/MS Lab GC Lab Standards Lab Radiological Lab	air conditioning
alcohols	vented storage cabinets	Organic Characterization Lab, Inorganic Extractions Lab Volatile Sample Preparation Lab GC/MS Lab GC Lab Standards Lab	air conditioning
inorganic chemicals	shelving cabinets	Organic Characterization Lab, Inorganic Extractions Lab, Electronics Shop, GC/MS Lab, GC Lab, Standards Lab Radiological Lab	air conditioning
trace metals	shelving cabinets	Inorganic Instrumentation Lab,	air conditioning
peroxides (H_2O_2)	vented storage cabinets	Inorganic Extractions Radiological Lab	air conditioning
acids	vented storage cabinets	Inorganic Extractions Organic Characterization Lab TCLP Lab	air conditioning

Table 18-1 (CONTINUED). Reagent Storage Requirements

Reagent	Storage	Location	Conditions
organic standards	explosion-proof refrigerators	Standards Lab Volatile GC/MS Semivolatile GC/MS	air conditioning
stock organic solvents	vented storage room	Solvent Storage Room	vented by fan 24 hr/day with explosion-proof lighting
stock inorganic chemicals (NaOH)	vented storage room vented storage cabinet	Solvent Storage Room Inorganic Sample Preparation	vented by fan 24 hr/day with explosion-proof lighting
radiochemical standards	lock limited access storage cabinet	Radiological Lab	air conditioning

Glossary

A

AAS: atomic absorption spectroscopy
accuracy: the nearness of a result or the mean of a set of results to the true or accepted result determined from compounds similar in chemical composition to analytes of interest added to every blank, sample, matrix spike, matrix spike duplicate, and standard. Recovery range is suggested rather than required (as opposed to surrogate standards).
aliquot: a measured portion of the whole sample that is used for analysis of the ion, element, or compound an analyst seeks to determine
analyte: any solution or media introduced into an instrument on which an analysis is performed, excluding calibration
analytical sample: any solution or media introduced into an instrument on which an analysis is performed, excluding calibration
anneal: a process of decontaminating glassware by heating and gradually cooling it
aqueous: similar to or dissolved in water
atomic absorption spectroscopy: measures the absorption of specific metallic elements at specific wavelengths

B

batch: a group of samples of similar matrix prepared at the same time in the same location using the same method
BFB: bromofluorobenzene
blank spike: a control sample of known composition that is processed by using the same analytical methods used on all other samples. Blank spikes serve as quality control checks.
BN: base/neutral
bromofluorobenzene: a compound used to establish mass spectral instrument performance for volatile analyses
BS: blank spike

C

calibration: the establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards
calibration check compounds: target compounds in the calibration check standard used to evaluate the calibration stability (precision) of the GC/MS system; must meet maximum percent difference criteria
calibration check standard: a standard used to determine the state of calibration of an instrument between periodic recalibrations
CCB: continuing calibration blank
CCC: calibration check compounds
CCV: continuing calibration verification
CERCLA: Comprehensive Environmental Response, Compensation, and Liability Act (1980); created a special tax that goes into a Trust Fund, known as Superfund, to investigate and clean up abandoned or uncontrolled hazardous waste sites

chain of custody: written documentation of who possessed a sample when; it includes both who relinquished and who received a sample. It tracks the location of the sample at all times.
characterization: a determination of the approximate concentration range of compounds of interest used to choose the appropriate analytical protocol
chromatogram: an electronic trace of the detector's response to a sample's components to which it is sensitive
chromatography: the separation of mixtures into their constituents by preferential absorption by a porous material, such as a column of silica or a strip of filter paper, or by a liquid
CL: control limit
class-S weights: used to calibrate balances; they are certified to be accurate within a certain range
CLP: Contract Laboratory Program
COC: Chain of Custody
continuing calibration blank (inorganics): a solution with no analyte. It is run at least every ten samples to ensure that no lab-introduced contamination has occurred.
continuing calibration verification (inorganics): the process of running an analytical standard with a known set of analytes at least every 10 analytical samples or every 2 hours, whichever is more frequent, to verify the calibration of the system
continuing calibration verification (organics): the process of running an analytical standard every 12 hours to verify the calibration of GC and GC/MS systems
contract laboratory program: supports the EPA's Superfund effort and provides a range of chemical analytical services on a high volume cost effective basis with legally defensible results
contract-required detection limit (inorganics): minimum level of detection acceptable under the applicable CLP Statement of Work
contract-required quantitation limit (organics): minimum level of detection acceptable under the applicable CLP Statement of Work; equal to the concentration of the lowest calibration standard analyzed for each analysis
control charts: basic tools for quality assurance that provide graphical means to demonstrate statistical control and to monitor a measurement process.
control limit: a range within which specified measurement results must fall to be compliant with QC criteria
CRDL: contract required detection limit
CRQL: contract required quantitation limit

D

day: unless otherwise specified, a calendar day
decafluorotriphenylphosphine: a compound used to establish mass spectral instrument performance for semivolatile analyses
detection limit: the smallest concentration/amount of some component that can be measured (by a single measurement) with a stated level of confidence
DFTPP: decafluorotriphenylphosphine
digestion log: an inorganics laboratory term; the official record of sample preparation (digestion)
DL: detection limit
duplicate (laboratory duplicate): a second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method; field duplicate is a second bottle used as a second aliquot of the same sample and usually unknown to the laboratory.

E

ECD: electron capture detector
EICP: extracted ion current profiles
ELCD: electrolytic conductivity detector; Hall detector
electrolytic conductivity detector: a detector used in the analysis of purgeable halocarbons
electron capture detector: a detector used for the analysis of chlorinated pesticides
elute: to remove by dissolving, as adsorbed material from adsorbent
external standard: a method of quantifying an unknown from a known in a separate source/sample
extractable: a compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas or liquid chromatography; includes semivolatile and pesticide/aroclor compounds
extracted ion current profile: instrument response to quantitation of selected ion masses at a specific retention time.

F

Fourier transform-infrared: a type of spectrometer in which radiation from an infrared source is passed through the sample and absorption occurs at certain wavelengths that are characteristic of particular chemical groups. It makes use of all frequencies from the source simultaneously rather than sequentially.
fragmentation: the molecular disruption that occurs when a molecule is bombarded by a field of electrons causing specific ion clusters or fragments to result; the fragmentation pattern (also known as mass spectrum) is used to identify a compound/molecule from a set of reference spectra.
FT-IR: Fourier Transform-infrared

G

gas chromatography: the physical separation of two or more compounds based on their differential distribution between two phases using an inert gas as the mobile phase
gas chromatography/mass spectrometry: mass spectrometer adds another dimension to GC data; GC is abundance vs. time, MS adds mass vs. time; the MS is the detector
GC: gas chromatograph(y)
GC/MS: gas chromatograph(y)/mass spectrometer(y)
good laboratory practices: guidelines established for CompuChem based on a set of rules, operating procedures, and sound practices established by the Food and Drug Administration in the *Federal Register* (volume 43, December 22, 1978) intended to ensure the quality and integrity of data generated by a laboratory

H

high performance liquid chromatography: a chromatographic technique wherein a liquid mobile phase transports a sample through a column containing a liquid stationary phase. Separation of sample components is based on preferential retention on the stationary phase.
holding time: the time specified by contract within which a sample must be extracted and/or analyzed from the time it was received (VTSR). However, for some non-CLP methods, holding times start at day of sampling instead of date of receipt. Different parameters have different maximum holding times as prescribed by the U.S. EPA Federal Register 40CFR136.
HPLC: high performance liquid chromatography

I

ICB: initial calibration blank
ICAP: inductively coupled argon plasma
ICP: inductively coupled plasma
ICV: initial calibration verification
IDL: instrument detection limit
inductively coupled (argon) plasma: a technique for the simultaneous or sequential multi-element determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Characteristic atomic line emission spectra are produced by excitation of the sample in a radio-frequency inductively coupled argon plasma.
in-house: at CompuChem Laboratories
initial calibration: the process of establishing a calibration curve
initial calibration blank: an inorganics term; reagent water with no analyte used to check for lab-introduced contamination and to set instrument background (zero the instrument). Cannot contain any analytes above the contract required detection limit.
initial calibration verification: when a standard with a known amount of analyte is run to check the calibration curve
instrument blank: used in volatile and pesticide analyses during each calibration period to verify that contaminants are not being introduced by components of the instruments or the laboratory
instrument calibration: analysis of a series of analytical standards of different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes
instrument detection limit: the lowest concentration of a compound that an instrument can detect
internal standard: in-house compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for VOAs), and sample extract (for semivolatiles and VOA medium level methanol extracts) at a known concentration, before analysis. Internal standards are used as the basis for quantitation of the target compounds. Used in volatile GC/MS and semivolatile GC/MS.

L

laboratory control sample: a sample of known composition. Aqueous and solid laboratory control samples are analyzed using the same sample preparation, reagents, and analytical methods employed for all other samples received. Control charts are generated from recovery of LCS components.
LCL: lower control limit
LCS: laboratory control sample
LIMS: laboratory information management system is a system of computer programs operated by a mainframe computer. It uses a system of analysis codes to schedule procedures and the QC samples required to be run with each batch of samples and to track a sample's progress through the laboratory.
lower control limit: mean % recovery - 3 x (standard deviation)

M

mass spectrum: a plot of the mass-to-charge ratio versus relative intensity of the ion current (also called a bar graph spectrum)
matrix: the predominant material of which the sample to be analyzed is composed; either water or soil/ sediment/sludge. Matrix is not synonymous with phase (liquid or solid).
matrix spike: aliquot of a sample fortified (spiked) with known quantities of specific compounds or analytes and subjected to the entire analytical procedure in order to indicate, by measuring recovery, the accuracy of the method for the matrix
matrix spike duplicate: a second aliquot of the same matrix as the matrix spike that is spiked in order to determine the precision of the method by calculating relative percent difference between the two
meniscus: the concave or convex upper surface of a column of liquid
method blank (inorganics): also referred to as blank, preparation blank or reagent blank. An analytical control that contains distilled, deionized water and reagents; it is carried through the entire analytical procedure (sample preparation and analysis).
method blank (organics): an analytical control consisting of all reagents and surrogate standards (where applicable), that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background contamination and efficiency of the extraction for the batch of samples with which it is prepared..
MS: matrix spike
MSD: matrix spike duplicate

N

National Bureau of Standards: now called National Institute of Standards and Technology
National Institute of Standards and Technology: a government agency that establishes standards for measurement
NIST: National Institute of Standards and Technology
NBS: National Bureau of Standards
NP Detector: Nitrogen phosphorus detector used for the specific detection of compounds containing either element

P

PCB: polychlorinated biphenyls
PE: performance evaluation; often used as "PE Sample"
PE sample: a sample sent by a client (or regulatory agency) who knows how much analyte it contains; used as a check of accuracy and precision of the entire analytical process and used to compare one lab's performance to another
photoionization detector: a detector that measures the current produced when a molecular species absorbs a photon of light energy and dissociates into the parent ion and an electron

PID: photoionization detector
ppb: parts per billion
ppm: parts per million
precision: the agreement or reproducibility of a set of replicate results among themselves or the agreement among repeat observations made under the same conditions; usually expressed as the standard deviation or percent difference
PT: proficiency testing
purge and trap: analytical technique used to isolate volatile (purgeable) organics by stripping/volatilizing the compounds from water or soil by a stream of inert gas, trapping the compounds on a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column

Q

QA: quality assurance
QC: quality control
quality assurance: a planned system of activities whose purpose is to provide assurance that the quality control program is actually effective
quality control: a planned system of controlled activities whose purpose is to provide a quality product

R

RCRA: Resource Conservation and Recovery Act (1976); a federal law that established a regulatory system to track hazardous substances from the time of generation to disposal
reagent water: water in which an interferent is not observed at or above the minimum quantitation limit of the parameters of interest
reconstructed ion chromatogram: a mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time
recovery: a determination of the accuracy of the analytical procedure made by comparing measured values for a spiked (fortified) sample against the known spike values. Recovery is determined by the following equation:

$$\%Rec = \frac{\text{Measured value}}{\text{spiked value}} \times 100\%$$

relative percent difference: a measure of precision; is calculated by the equation below and is always expressed as the absolute value or zero:

$$RPD = \frac{\text{difference}}{\text{average}} \times 100$$

relative response factor: a measure of the relative mass spectral response of an analyte compared to its internal standard
relative standard deviation: a statistical indicator of the amount a measured value differs from the actual value; calculated by the equation :

$$RSD = \frac{\text{Standard deviation}}{\text{Mean}} \times 100$$

RIC: reconstructed ion chromatogram
RPD: relative percent difference

RRF: relative response factor
RSD: relative standard deviation
run: a continuous analytical sequence consisting of prepared samples and all associated quality control measurements as required by the method or contract Statement of Work; the analysis, or single "run," of a prepared sample on an analytical instrument

S

sample: a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number
sample custodian: the individual assigned responsibility for custody of samples and access to the locked refrigerators in which the samples are stored
sample delivery group: defined by the following, whichever is most frequent:

- each case of field samples received, or
- each 20 field samples within a case, or
- each 14 calendar day period (7-calendar day period for 14-day data turnaround contracts) during which field samples in a case are received (beginning with the receipt of the first sample in the sample delivery group).

Sample Management Office: an agency contracted by EPA to manage the financial and administrative duties of the Contract Laboratory Program
SampleSaver: a patented container for collection of environmental samples and shipment of them to CompuChem Environmental Corporation.
sample spike: another term for matrix spike
SDG: sample delivery group
semivolatile compounds: acid and base/neutral compounds amenable to analysis by extraction of the sample with an organic solvent; usually have a ring structure and are found in, for example, coal tars
septum: a dividing wall or membrane
SMO: Sample Management Office
SOP: Standard Operating Procedure
SOW: statement-of-work
sparge: to introduce air or other gas such as nitrogen into a liquid with the intent of volatilizing dissolved species
SPCC: system performance check compounds
spike: the addition of a known amount of analyte or compound to a sample or matrix
standard analysis: an analytical determination made with known quantities of target compounds; used to determine response factors
standard operating procedure: a document that describes laboratory procedures (analytical and non-analytical) and serves as a training aid and reference tool
Statement of Work: a compilation of Contract Laboratory Program procedures issued by the EPA to be followed for sample receipt and handling, analytical methods, data reporting and deliverables, and document control
surrogate standard: brominated, fluorinated, or isotopically labeled compounds added to blanks, standards, matrix spikes,and matrix spike duplicates to evaluate extraction efficiency by measuring recovery. Generally

applicable to volatile, semivolatile, and pesticide/aroclor analyses.
system monitoring compounds: brominated or deuterated compounds added to every blank, standard, sample, matrix, and matrix spike duplicate to evaluate the performance of the entire purge and trap -- gas chromatograph -- mass spectrometer system (3/90 volatiles only)
system performance check compounds: target compounds in the calibration standards designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Must meet minimum response factor criteria.

T

target compound list: a list of compounds designated by the Statement of Work for analysis
TCL: target compound list
tentatively identified compounds: compounds detected in samples that are not target compounds, internal standards, system monitoring compounds or surrogates; also known as extraneous compounds
TIC: tentatively identified compounds
TOC: total organic carbon
total organic carbon: tests for organic carbons, in water and wastewater
total organic halides: tests for compounds containing chlorine, iodine, and bromine atoms
total petroleum hydrocarbons: tests for long chain carbons found in, for instance, fuels, gas, and oils
TOX: total organic halides
TPH: total petroleum hydrocarbons
TR: traffic report
traffic report: an EPA sample identification form filled out by the sampler which accompanies the sample during shipment to the laboratory and documents sample condition and receipt by the laboratory

U

UCL: upper control limit
United States Environmental Protection Agency: federal agency created to consolidate federal authority over pollution; is given the power to administer programs that address the environmental problems of air and water pollution, pesticides, toxic substances, radiation, noise, and solid waste management
upper control limit: mean percent recovery + 3 x (standard deviation)
U.S. EPA: United States Environmental Protection Agency

V

validated time of sample receipt: the date on which a sample is received by CompuChem, as recorded on the shipper's delivery receipt and Sample Traffic Report
VOA: volatile organic analysis
volatile compounds: compounds amenable to analysis by the purge and trap technique
VTSR: validated time of sample receipt

Sources

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INTERNATIONAL TECHNOLOGY CORPORATION

Analytical Services Division

Operation-Specific

Quality Assurance Management Plan

INTERNATIONAL TECHNOLOGY CORPORATION

March 24, 1994

Mr. E. Raymond Kruse
Subcontract Administrator
Ensafe/Allen & Hoshall
5720 Summer Trees Drive
Memphis, TN 38134

Dear Mr. Kruse,

Please sign the attached form to achknowledge that you have recieved your controlled copy of the ITAS Operation Specific Quality Assurance Plan Revision 0-Procedure change forms. Thank you.

Controlled Copy No.: KN-OSQAMP-001

Please return the attached form to:

Chris Rigell
Quality Assurance Manager,
IT Analytical Services
5815 Middlebrook Pike
Knoxville, TN 37921

ITAS-KNOXVILLE LABORATORY

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Documents Issued	**Documents Superseded**
Number, Revision, and Title	**Number, Revision, and Title**
Operation Specific Quality Assurance Management Plan Revision 0 -Procedure change forms OSQAMP-1 pages 243/244 OSQAMP-2 page 165 OSQAMP-3 page 241 OSQAMP-4 Table 8.2-1 page 133 OSQAMP-5 Table 8.8-1 page 161 OSQAMP-6 TABLE 8.5-1 page 198 pages 77, 81, and 163 Appendix N Navy Specific Attachment pages 1-6 Glossary pages 1-18	OSQAMP Revision 0 issued October 13, 1993.

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Copyholder's Signature: E Ray Kruse **Date:** 23 APR 94

INTERNATIONAL
TECHNOLOGY
CORPORATION

October 13, 1993

E. Raymond Kruse
Subcontract Administrator
Ensafe/Allen & Hoshall
5720 Summer Trees Drive
Memphis, Tennessee 38134

Dear Mr. Kruse:

Enclosed please find the Operation Specific Quality Assurance Management Plan with the Knoxville Laboratory Appendix for IT Analytical Services Knoxville Laboratory. Also included are additional appendix dividers should you need the facility specific information for the other laboratories within IT Analytical Services.

Please sign the attached acknowledgement letter and return it to me for my distribution files.

If you have any questions or need any other information call me at (615) 588-6401.

Sincerely,

Christopher Rigell
Quality Assurance Manager

ITAS-KNOXVILLE LABORATORY

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Verified by Ray Kruse

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Operation-Specific

Quality Assurance Management Plan

IT ANALYTICAL SERVICES DIVISION

Revision 0

September 1, 1993

Operation-Specific
QUALITY ASSURANCE MANAGEMENT PLAN
IT ANALYTICAL SERVICES DIVISION

KNOXVILLE LABORATORY

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LIST OF ACRONYMS

AA	Atomic Absorption
ANSI	American National Standards Institute
AR/COC	Analysis Request/Chain-of-Custody
AS	Analytical Spike
ASTM	American Society for Testing and Materials
BFB	Bromofluorobenzene
BLK	Blank
BS	Blank Spike
BSD	Blank Spike Duplicate
CCC	Continuing Calibration Compounds
CCS	Continuing Calibration Standard
CEO	Chief Executive Officer
CF	Calibration Factor
CHP	Chemical Hygiene Plan
CLP	Contract Laboratory Program
COC	Chain-of-Custody
CRDL	Contract Required Detection Limit
CRQL	Contract Required Quantitation Limit
CS	Check Standard
CUR	Condition Upon Receipt
CVAA	Cold Vapor Atomic Absorption
DFTPP	Decafluorotriphenylphosphine
DOC	Dissolved Organic Carbon
DOT	Department of Transportation
DQO	Data Quality Objective
DRC	Data Review Checklist
EDT	Electronic Data Transfer
EMSL-LV	Environmental Monitoring Laboratory-Las Vegas
EPA	U. S. Environmental Protection Agency
FAS	Field Analytical Services
FB	Field Blank
FD	Field Duplicate
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
GFAA	Graphite Furnace Atomic Absorption Spectroscopy
HDPE	High Density Polyethylene
HPLC	High Performance Liquid Chromatography
HRGC	High Resolution Gas Chromatography
HRMS	High Resolution Mass Spectrometry

LIST OF ACRONYMS (continued)

HTV	Holding Time Violation
IATA	International Air Transportation Association
ICAO	International Civil Aviation Organization
ICP	Inductively Coupled Argon Plasma Spectroscopy
ID	Identification
IR	Infrared Spectroscopy
IS	Internal Standard
ISO	International Standard Organization
IT	IT Corporation
ITAS	IT Analytical Services
JPR	Job Performance Review
LCL	Lower Control Limit
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
LRMS	Low Resolution Gas Chromatography
LST	Laboratory Safety Training
LWL	Lower Warning Limit
MB	Method Blank
MBAS	Methylene Blue Active Substance
MDA	Minimum Detectable Activity
MDL	Method Detection Limit
MOP	Manual of Practice
MS	Matrix Spike
MSA	Method of Standard Additions
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheet
NBS	National Bureau of Standards
NCM	Nonconformance Memo
ND	Not Detected
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards Technology
NMOC	Non-Methane Organic Compounds
NPDES	National Pollutant Discharge Elimination System
NRC	Nuclear Regulatory Commission
OS-QAMP	Operation-Specific Quality Assurance Management Plan
PAH	Polynuclear Aromatic Hydrocarbons (or PNA)
PCB	Polychlorinated Biphenyls
PE	Performance Evaluation
PPE	Personal Protective Equipment
PQL	Practical Quantitation Limit

LIST OF ACRONYMS (continued)

PUF	Polyurethane Foam
QA	Quality Assurance
QAMP	Quality Assurance Management Plan
QAPjP	Quality Assurance Project Plan
QAPP	Quality Assurance Program Plan
QA/QC	Quality Assurance/Quality Control
QA/QCC	Quality Assurance/Quality Control Coordinator
QAS	Quality Assurance Summary
QC	Quality Control
RB	Reagent Blank
RCRA	Resource Conservation Recovery Act
RF	Response Factor
RPD	Relative Percent Difference
RRF	Relative Response Factor
RSD	Relative Standard Deviation
RSO	Radiation Safety Officer
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SPCC	System Performance Check Compounds
SRM	Standard Reference Material
SS	Surrogate Standard
TAT	Turnaround Time
TB	Trip Blank
TCLP	Toxicity Characteristic Leaching Procedure
TKN	Total Kjeldahl Nitrogen
TOC	Total Organic Carbon
TOX	Total Organic Halogens
TQM	Total Quality Management
UCL	Upper Control Limit
UN	United Nations
USEPA	United States Environmental Protection Agency
UWL	Upper Warning Limit
VOA	Volatile Organic Analytes
VOST	Volatile Organic Sampling Train
WS	Water Supply
WP	Water Pollution

APPENDIX LIST

APPENDIX LIST (continued)

APPENDIX LIST (continued)

1.0 MANAGEMENT COMMITMENT AND ORGANIZATION

1.1 Statement of Management Position on Quality

IT Corporation (IT) is committed to providing quality services for environmental management; services which meet the needs of our clients, satisfy regulatory requirements, and are commensurate with the current state of the art. To satisfy our clients' quality objectives, to meet regulatory requirements, and to comply with IT corporate-wide requirements, IT Analytical Services (ITAS) Division has adopted a comprehensive Quality Assurance (QA) Program. The principles and practices of the Program apply to every Associate at every level within ITAS; they are fundamental to the way we do business and to the services we provide.

The Quality Assurance Management Plan (QAMP) is an overall statement of Program policy. This Plan provides guidance to ITAS Associates in fulfilling their responsibilities and serves as a statement to external parties of ITAS' commitment to quality.

The ITAS QAMP recognizes that some QA activities must be detailed and adopted on an operation-specific basis. To document individual laboratory QA practices, the ITAS QAMP requires the preparation of this Operation-Specific QAMP.

Implementation of the QA Program is the responsibility of all ITAS Associates. Management at every level has the commitment, duty, and authority to lead the development and implementation of a structured management system that provides the framework to support the QA Program. Management will assure that the principles and practices of the QA Program are implemented and followed.

A Quality Assurance/Quality Control Coordinator (QA/QCC) is assigned to each ITAS operating unit to verify that the QA Program is implemented as intended by the Associates performing the work on a daily basis. The QA/QCC has the authority and duty to stop work if and when necessary to satisfy QA Program requirements.

To verify that the QA Program is successfully implemented at each ITAS operating unit, independent assessments are directed or conducted by the Division Director, Quality Assurance/Quality Control. In addition, operating units are subject to assessments by the Vice President, Quality and Health Services, and by various regulatory authorities and other

outside agencies.

1.2 IT Analytical Services (ITAS) Division Organizational Structure

1.2.1 ITAS Operating Units

The ITAS Division consists of twelve operating units at the following locations:

- ITAS-Austin Laboratory (Austin, Texas)
- ITAS-Cerritos Laboratory (Cerritos, California)
- ITAS-Cincinnati Laboratory (Cincinnati, Ohio)
- ITAS-Edison Laboratory (Edison, New Jersey)
- ITAS-Knoxville Laboratory (Knoxville, Tennessee)
- ITAS-Oak Ridge Laboratory (Oak Ridge, Tennessee)
- ITAS-Pittsburgh Laboratory (Pittsburgh, Pennsylvania)
- ITAS-Special Analysis Laboratory (Knoxville, Tennessee)
- ITAS-St. Louis Laboratory (St. Louis, Missouri)
- ITAS-Richland Laboratory (Richland, Washington)
- ITAS-Field Analytical Services-West (Martinez, California)
- ITAS-Field Analytical Services-East (Knoxville, Tennessee)

ITAS is a laboratory network dedicated to the analysis of hazardous, radioactive, and mixed waste material. Radiochemical analyses are performed at the ITAS laboratories located in Oak Ridge, Richland, and St. Louis. Field Analytical Services (FAS) groups, located in California and Tennessee, serve as a key link between field operations and the analytical laboratories.

1.2.2 Organization Charts

The organizational structure for the ITAS Division is displayed in Figure 1.2-1. The organizational structure for each ITAS operating unit is displayed in Appendix Section 1. The quality-related responsibilities of the members of the ITAS Division and of the ITAS operating units are outlined in Section 1.3.

1.2.3 Equipment

A listing of instrumentation used by each ITAS operating unit is shown in Appendix Section 2.

1.2.4 Facility Security

Each ITAS laboratory is a limited access, secure facility. To ensure that only authorized personnel are able to enter the building from an entrance that is not

monitored. entry into each building is limited in one or more of the following ways at a minimum:

- The use of key pads or electronic locks activated by swipe or magnetic cards which are issued only to authorized personnel
- Locking doors and issuing keys only to authorized personnel
- During business hours, entry is possible only through the main entrance. This entrance is monitored at all times by the receptionist. All guests are required to sign in using the visitor logbook and obtain a visitors badge.
- Alarm systems to detect unauthorized entrance

1.3 Quality Organization

The achievement of quality in all activities is the responsibility of each ITAS Associate. Quality related responsibilities within the operational unit provide for the implementation of the QA Program and completion of quality control (QC) activities.

The quality related responsibilities in IT and the ITAS Division are described in the following sections. The quality-related responsibilities may be reassigned by dividing the activities among different individuals or enhanced by adding activities, but they may not be eliminated.

1.3.1 Vice President, Quality and Health Services, IT Corporation

- Reports directly to the Chief Executive Officer (CEO), IT Corporation
- Approves the ITAS QAMP and the ITAS Operation-Specific QAMP
- Provides independent QA review by participating in or conducting assessments of ITAS operations
- Participates in finding solutions to quality problems not readily resolved within ITAS

1.3.2 Vice President, IT Analytical Services, IT Corporation

- Reports directly to the CEO, IT Corporation
- Approves the ITAS QAMP, the ITAS Operation-Specific QAMP, and ITAS Manuals of Practice (MOP)
- Assumes the ultimate responsibility for the QA Program within ITAS operations
- Assigns specific quality-related responsibilities within the operating units to the ITAS Laboratory and FAS Directors
- Periodically determines the effectiveness of the QA Program, recommending changes to the

Division Director, QA/QC

1.3.3 *Division Technical Director, IT Analytical Services*

- Reports directly to the Vice President, IT Analytical Services
- Maintains and disseminates as appropriate to the laboratories current information on regulations and approved methodologies performed by ITAS laboratories
- Acts as a technical consultant, interfacing with the Division Director, QA/QC for quality related issues to assure uniform technical excellence across ITAS operations
- Provides guidance and training to all Laboratory Technical Directors
- Approves the ITAS QAMP, the ITAS Operation-Specific QAMP and appendices, and ITAS MOPs. Also reviews other quality and technical documents produced by ITAS for accuracy, completeness, and applicability to relevant technical goals, regulations, and methodologies
- Guides implementation of the QA Program through training programs

1.3.4 *Division Director, Quality Assurance/Quality Control, IT Analytical Services*

- Reports directly to the Division Technical Director with a QA "dotted line" responsibility to both the Vice President, IT Analytical Services and the Vice President, Quality and Health Services, IT
- Reviews and approves ITAS QA documents
- Approves the ITAS QAMP, the ITAS Operation-Specific QAMP and appendices, and ITAS MOPs
- Provides guidance and training to all laboratory QA/QCCs
- Oversees independent assessments (audits) of ITAS laboratories to identify areas where improvement is needed to comply with the QA Program
- Verifies completion of corrective actions required to correct nonconformances identified during assessments
- Acts as the focal point for improvements and changes to the QA Program, approves and initiates these changes
- Discusses unresolved nonconformances identified during assessments or brought to the Director's attention by the QA staff for resolution with the ITAS Operational Directors, Vice President, IT Analytical Services and/or Vice President, Quality and Health Services, IT
- Suspends further processing in operational units that are out-of-control until the nonconformance is

corrected

1.3.5 Division Operations Director, IT Analytical Services

- Reports directly to the Vice President, IT Analytical Services
- Approves the ITAS Operation-Specific QAMP
- Assumes responsibility and provides resources for implementation of the QA Program within the operational units
- Assigns specific quality-related responsibilities within the operational units to resolve problems
- Periodically determines the effectiveness of the QA program

1.3.6 Field Analytical Services Director

- Reports directly to the Vice President, IT Analytical Services with operational responsibility to the Division Operations Director
- Implements the QA Program within the operation
- Approves quality related documents
- Periodically determines the effectiveness of the QA Program within the operation
- Responsible for the issuance of analysis reports for the operation
- Maintains adequate staffing documented on organization charts

1.3.6.1 Field Analytical Services Business Unit/Office Manager

- Reports directly to the FAS Director
- Implements the QA Program within the business unit/office
- Reviews and approves quality related documents
- Conducts project reviews to assure compliance with QA Programs
- Chairs the business unit/office Quality Team
- Coordinates performance of client satisfaction surveys with client QA Officer
- Approves the ITAS Operation-Specific QAMP and the applicable appendix

1.3.6.2 FAS Project Manager

- Directs preparation of quality related documents for projects
- Designs project QA programs to meet project objectives
- Establishes analytical project data quality objectives
- Coordinates with the laboratory to ensure that quality issues are addressed at all stages of a project

- Assures that project quality requirements are met

1.3.6.3 FAS Field Analytical Specialist

- Prepares project quality documents
- Implements project quality requirements for task performance and documentation
- Prepares and maintains records documenting quality control problems implemented for a project
- Adheres to the ITAS QA Program for governing project activities unless superceded by a project/program-specific plan

1.3.7 Laboratory Director (Operating Unit Director)

- Reports directly to the Vice President, IT Analytical Services with operational responsibility to the Division Operations Director
- Implements the QA Program within the laboratory
- Approves quality related documents
- Approves the ITAS Operation-Specific QAMP and the appropriate appendix
- Periodically determines the effectiveness of the QA Program within the operation
- Maintains adequate staffing documented on organization charts

1.3.7.1 Systems Manager

- Reports directly to the Laboratory Director
- Supervises daily activities of the Project Management, Laboratory Information Management System (LIMS) Operation, Sample Control, Administrative, and Report/Proposal Production Groups
- Oversees the log-in of all samples received, completion of chain-of-custody records, and initiation of project records
- Supervises sample storage facilities
- Works with Operations Manager and/or Group Leaders to assure the requirements of the project are met in a timely manner
- Supervises the verification of software for data processing
- Ensures compliance with QA Program within the laboratory

1.3.7.2 Operations Manager

- Reports directly to the Laboratory Director
- Supervises daily activities of the Operational Groups
- Supervises QC activities performed as a part of routine analytical operations

- Supervises the preparation and maintenance of laboratory records
- Oversees the preventive maintenance program
- Supervises laboratory participation in interlaboratory accreditation and proficiency programs
- Ensures compliance with QA Program within the laboratory
- In the absence of a Group Leader, assumes their quality responsibilities

1.3.7.3 *Quality Assurance/Quality Control Coordinator*

- Reports directly to the Laboratory Director or the Laboratory Technical Director as defined by the organizational chart
- Develops and defines the Total Quality Program for the laboratory within Division and Corporate guidelines
- Monitors laboratory practices to ensure conformance with the ITAS QA Program, regulatory requirements and client specified contractual obligations
- Recommends resolutions for ongoing or recurrent nonconformances within the laboratory
- Shuts down out-of-control analyses or laboratory groups if necessary until the nonconformance is corrected and notifies the appropriate Laboratory Director and/or the Division Director, QA/QC
- Reviews data quality measures, including statistical data to verify that the laboratory is meeting stated quality goals
- Closes findings and observations of QA audits
- Performs two QA Systems Audits per year (one full systems audit and one follow-up audit)
- Establishes and supervises laboratory QA training programs
- Assists in the preparation of and approves Quality Assurance Project Plans (QAPjPs)
- Serves as the focal point for the reporting and disposition of nonconformances
- Performs monthly surveillances of the laboratory (except during months when a systems audit or follow-up audit is performed)
- Maintains certification and accreditation programs
- Arranges for insertion of QC samples into the laboratory sample stream and reviews the results
- Performs statistical analyses utilizing results of the QC sample analyses and reviews the data to assure

compliance with stated quality objectives

- Assists in the performance of QA Systems Audits
- Maintains controlled documents
- Prepares monthly quality report to management
- Performs Client Satisfaction Surveys

1.3.7.4 Technical Director

- Reports directly to the Laboratory Director
- Provides technical overview of laboratory activities
- Establishes and supervises the training of analysts in good laboratory practices and analytical methodologies
- Writes and reviews Standard Operating Procedures (SOPs), assuring compliance with regulations and approved methodology
- Serves as technical consultant to Project Management and analytical staff in choosing the correct methods, QC and analytical techniques
- Evaluates analytical techniques, procedures, and instrumentation and provides recommendations for improvement to the Laboratory Director
- Recommends standards for purchasing instrumentation, equipment, reagents, gases, and chemicals in accordance with SOPs and approved methodologies
- Defines the calibration and preventive maintenance programs
- Approves customer requested variances to methods
- Responsible for specific Division SOPs and method improvement variances
- In some laboratories, supervises the QA/QCC as defined by the organizational chart
- Ensures compliance with QA/QC Program within the laboratory

1.3.7.5 Group Leader/Team Leader

- Reports directly to the Operations Manager or the Laboratory Director in the absence of an Operations Manager
- Serves as the lead analyst within the group (e.g. GC/MS, GC, AA/ICP, General Chemistry, etc.) and supervises daily activities of all other analysts within their group
- Organizes and schedules the analytical testing program with consideration for sample holding times
- Supervises QC activities performed as a part of routine analytical

operations

- Implements data verification procedures
- Supervises the preparation and maintenance of laboratory records
- Evaluates instrument performance and supervises the calibration and preventive maintenance programs
- Oversees or performs review and approval of all analytical data and submits it to the Project Manager for reporting
- Reports out-of-control or nonconforming situations to the appropriate managers (e.g. Operations Manager, Laboratory Director, Technical Director, and/or Project Manager) and the QA/QCC.
- Supervises maintenance of instruments and scheduling of repairs
- Ensures compliance with QA Program within the laboratory

1.3.7.6 Project Manager

- Monitors analytical and QA project requirements
- Assists the Systems Manager, QA/QCC, and Technical Director with interpretation of work plans or QAPjP requirements
- Assists the Systems Manager in establishing goals, priorities, schedules, and work assignments
- Keeps the laboratory and the client informed of project status
- Approves customer requested variances to methods
- Monitors, reviews, and evaluates the progress and performance of projects
- Reviews data packages for completeness and compliance to client needs
- Prepares Quality Assurance Summaries (QASs)
- Generates and signs analytical reports (or designee)

1.3.7.7 Analyst

- Implements the QA Program within the laboratory
- Performs analytical procedures and data recording in accordance with accepted methods
- Performs and documents calibration and preventive maintenance (may also be performed by a calibration control group)
- Performs data processing and data verification
- Immediately reports out-of-control or nonconforming situations to the Group Leader and QA/QCC

- Maintains control charts or tables

1.3.7.8 *Sample Custodian*

- Ensures implementation of proper sample receipt procedures, including maintenance of chain-of-custody
- Logs samples into the LIMS system
- Ensures that all samples are stored in the proper environment
- Assists Waste Management staff with sample disposal

1.3.7.9 *Document Control Coordinator*

- Receives and initiates chain-of-custody for data packages received from the laboratory
- Maintains custody of data packages

1.3.7.10 *Data Reporting Staff*

- Accurately transfers data from verified laboratory data packages or laboratory report forms to Certificates of Analyses or other deliverables
- Performs a thorough QA review of all Certificates of Analysis or final reports to verify the accurate transcription of data
- Prepares electronic data transfers (EDTs)

2.0 QUALITY ASSURANCE PROGRAM DESCRIPTION

2.1 Introduction

IT has defined Quality as "meeting the requirements of our clients, both internal and external". To achieve Quality, a Total Quality Management (TQM) process has been established and is being implemented throughout the company. Part of this implementation is through Division QA Programs.

It is the purpose of the ITAS QA Program, as expressed in the QAMP, to provide data which are of known and acceptable quality. To achieve this, a system is described which controls the following:

- Preservation of samples
- Receipt and handling of samples
- Processing and analysis of samples
- Analytical equipment
- Data verification
- Data reporting
- Records management
- Management review

ITAS recognizes that all laboratory and field Associates affect data quality. This Plan has been prepared so that all IT Associates will be cognizant of the policies adopted by ITAS for the production of analytical data and will be aware of their responsibilities. Specific implementation instructions for quality practices are documented in SOPs for each ITAS operating unit.

As cross referenced in Table 2.1-1, the ITAS QA Program meets the basic requirements of the following references:

- Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80, Office of Monitoring Systems and Quality Assurance, Office of Research and Development, United States Environmental Protection Agency, EPA 600/4-83-004, February 1983
- Quality Assurance Program Requirements for Environmental Programs, American Society for Quality Control, Energy Division, Environmental Waste Management Committee, ANSI/ASQC-E4-19xx (Formerly EQA-1), July 1992
- Quality Assurance Program Requirements for Nuclear Facilities, The American Society of Mechanical Engineers, ANSI/ASME NQA-1-1989 edition

- Quality Assurance, Office of Nuclear Energy & Office of Environmental Safety and Health, United States Department of Energy, DOE ORDER 5700.6C, August 1991

- Performance Criteria for Radiobioassay, ANSI N 13.30, September 1989

- Measurement Quality Assurance for Radioassay Laboratories, ANSI N 42.2, Revised May 21, 1992, Revision 10A

- Quality Systems-Model for Quality Assurance in Design/Development, Production, Installation, and Servicing, ISO 9001 (ANSI/ASQC Q91-1987)

Additional guides utilized by the radiochemistry laboratories include:

- USNRC Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operation) - Effluent Streams and the Environment

- ASTM Standard Guide C 1009 - 83. Establishing a Quality Assurance Program for Analytical Chemistry Laboratories within the Nuclear Industry

For the purposes of this Plan, the ITAS Quality System is composed of QA and QC activities. The terms are defined and used as follows:

- Quality System - "The collective plans, activities, and events that are provided to ensure a product, process, or device will satisfy given needs." ***ANSI/ASQC Standard A3***

- Quality Assurance - "All those planned or systematic actions necessary to provide confidence that a product or service will satisfy given needs."***ANSI/ASQC Standard A3***

- Quality Control - "A process which measures actual quality performance, compares with standards, and acts on the difference!" ***Juran, 1974***

2.2 Objectives of the QA Program

The overall objective of the QA Program for ITAS operations is to provide data of known quality that meet client requirements. In general, to accomplish this, each laboratory must:

- Maintain an effective, ongoing QC Program to measure and verify laboratory performance

- Meet data requirements for accuracy, precision, and completeness through the use of proven methodologies

- Provide sufficient flexibility to allow controlled changes in routine methodology to meet specific data requirements

- Monitor operational performance of the laboratory on a routine basis and provide corrective action as needed

- Recognize and promptly correct for any

factors which adversely affect quality

- Maintain complete records from sample submittal through laboratory analysis, data verification, reporting, and sample disposal

In order to meet these objectives, two levels of management controls are required. Controls at the organizational level include all activities that support common or standardized functions such as Associate qualifications and training, document control, and material procurement. Controls at a project level consist of the project-specific QA program activities necessary to produce the desired type and quality of product.

2.3 *Quality Assurance Documents*

Table 2.3-1 summarizes the ITAS QA documents, the purpose of each, and the required approvals for each. Document control, distribution, and revision is discussed in Section 5.

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3.0 ASSOCIATE TRAINING AND QUALIFICATION

All quality related activities performed by ITAS shall be accomplished by Associates qualified on the basis of education, experience, and training. The following definitions are relevant to the discussion of training in this section:

- Training - In-depth instruction to develop proficiency in the application of requirements, methods, and procedures. Such instruction may be internal or external classroom sessions, courses, or on-the-job assignments.
- Indoctrination - To instruct in fundamentals so as to provide understanding of principles involved.
- Qualification (Personnel) - The characteristics or abilities gained through training or experience or both, that enable an individual to perform a required function.
- Certification - The action of determining, verifying, and attesting, in writing, to the qualifications of personnel (Associates) or material.
- Orientation - The act or process of acquainting individuals with the existing situation, environment, or condition.

3.1 Associate Qualifications

Each operating unit shall have job descriptions for all positions. These job descriptions shall specify the minimum qualifications in terms of education and experience, knowledge, and skills necessary for an Associate to carry out work. The operational supervisors shall compare each Associate's performance with the qualifications established in his/her job description at least annually. This should be done in conjunction with the Associate's Job Performance Review (JPR).

ITAS normally expects necessary knowledge and fundamental chemical laboratory skills to have been demonstrated by formal academic training to include course work in general chemistry, qualitative analysis, quantitative analysis, and instrumental analysis. Qualifications of all professional Associates shall be documented by resumes which include academic credentials, employment history, experience, and professional registrations. A copy of a current resume shall be included in each Associate's training file. When applicable, each professional Associate shall be formally qualified to perform their lab functions by their supervisor. Applicability is dependent upon the Associate's job function. Associates must be formally qualified if their work involves sample management, sample preparation, clean-up, or analysis

procedures, or project management. Record of personnel qualification must be documented in the Associate's training file. An example form is shown in Figure 3.1-1.

3.2 Orientation and Training of Laboratory Staff

Training is performed to maintain proficiency, promote improvement, and to stimulate professional development. ITAS staffs include professional Associates who are scientists. Such Associates shall be assigned duties within the capabilities of their education and experience by the appropriate supervisor and shall be qualified to perform and train others on specified procedures based on this experience.

ITAS Associates are qualified through indoctrination and experience which is documented in resumes and training files. Each new Associate shall be supervised in their activities by experienced Associates until, in the opinion of the supervisor, they are capable of independently performing their duties. This authorization to perform independently shall be documented in the training files on a personnel qualification form. In addition, training for management Associates shall include professional, managerial, communication, and interpersonal skills. On-going or periodic assessments will be performed to determine training needs and effectiveness of instruction.

An individual training record (example shown in Figure 3.2-1) shall be maintained for each Associate. This form must be reviewed and updated, as necessary, by the Associate on an annual basis.

3.2.1 Quality Assurance Orientation

Each newly hired ITAS Associate is required to go through QA orientation. The QA/QCC shall conduct this orientation in accordance with SOPs within two weeks of the Associate's report-to-work date. The QA/QCC shall review the following topics (at a minimum) with the new Associate:

- Overview of ITAS QA Program
- ITAS philosophy on data integrity and meeting client requirements
- ITAS QA documents
- Pertinent regulatory QA requirements
- Data recording practices
- Nonconformance and corrective action
- Chain-of-Custody procedures

An example form used for documenting QA orientation is shown in Figure 3.2-2.

Following QA orientation, the Associate is required to take a written QA examination

to demonstrate an understanding of the laboratory QA program and to determine if any areas covered require further training.

3.2.2 Quality Assurance Training

Training in the nature and goals of the QA Program shall be provided at least once a year to all laboratory Associates. Formal training sessions will be conducted and documented by the QA/QCC. The training program shall address regulatory requirements as appropriate, basic QC practices, responsibilities of the technical staff, responsibilities of the QA/QCC, the reporting of nonconformances, and the performance of audits. In addition, each laboratory Associate shall become familiar with the laboratory QA Program by reading pertinent sections of the ITAS QAMP and the Operation-Specific QAMP, and QC procedures appropriate to his/her position.

3.2.3 Health and Safety Orientation

Each newly hired ITAS Associate, contract worker, or working visitor is required to go through a health and safety orientation before they are allowed to work in the laboratory (as per the laboratory Chemical Hygiene Plan). This orientation is to be performed as soon as possible after or within one week of the Associate's report-to-work date. The Associate's immediate supervisor or the laboratory Health and Safety Coordinator should perform this orientation, which includes viewing videotapes on the OSHA Laboratory Standards and "basic" laboratory safety. The Associate will receive information on the following:

- Safety Policy and Program
- Laboratory Emergency (Contingency) Plan
- Safety Equipment
- Material Safety Data Sheets (MSDSs) (content and location)

The supervisor will supply basic job-related information on:

- Chemical Safety
- Electrical Safety
- Thermal Safety
- Mechanical Safety
- Waste Disposal

All health and safety orientations will be documented.

3.2.4 Health and Safety Training

Health and safety training at ITAS is required for all Associates, contracted workers, and temporary help. This training is to be given within 90 days of the start-to-work date and will be provided by Division Health and Safety or by qualified instructors in the laboratories.

Documentation is maintained in the Associate's training records.

ITAS health and safety training includes the following:

- Laboratory Safety Training (LST)- required for each Associate every two years, at a minimum
- Chemical Hygiene Plan (CHP) Training- each Associate is required to attend an annual refresher

3.2.5 QA/QCC Training

All QA/QCCs shall receive training from the Division Director, QA/QC, so that they are proficient in the requirements of the ITAS QA Program and it's application. Formal training, directed by the Division Director, QA/QC, is performed on an annual basis. Continued proficiency of QA/QCCs shall be maintained through active participation in QA audits and the preparation and review of QA documents. Evaluation of QA/QCC proficiency shall be conducted on an annual basis by the Division Director, QA/QC, as secondary reviewer of the QA/QCC's JPR.

3.2.6 Lead Auditor Certification

ITAS has developed a program for certifying Lead Auditors. QA/QCCs are the primary participants in this program which is described in the ITAS System Procedure No. 8907-QAC-04, "Standard Certification at ITAS Laboratories." All internal ITAS systems audits are lead by an ITAS certified Lead Auditor. Lead Auditor certification is documented with an example form shown in Figure 3.2-3 and with a certificate signed by the Division Director, QA/QC.

3.3 Training Records

Each ITAS Associate has an individual training file maintained by the QA/QCC. The documents included in the training file are the following:

- Associate's resume (current and in ITAS format)
- Individual training record
- Reference to qualifying sample sets
- QA examinations
- Professional certificates (copies)
- Attendance records of training courses
- Personnel qualification records

Each Associate shall review their training file annually, at a minimum, to ensure completeness and correctness of contained information.

4.0 PROCUREMENT OF ITEMS AND SERVICES

This chapter defines the ITAS requirements for the procurement of items and services. This program will provide for:

- Assurance that purchased items and services meet established requirements and perform as expected
- Evaluation and selection of vendors
- Inclusion of applicable technical and administrative requirements in procurement documents

4.1 Selection of Vendors

Prospective vendors will be evaluated and selected based on the following criteria as appropriate:

- Evaluation of the vendor's history of providing an identical or similar product which performs satisfactorily in actual use
- Objective evaluation of the vendor's current quality records supported by documentation
- Direct audit of the vendor's technical and quality capability

The QA/QCC shall determine the needed level of qualification based on the importance of the item or service being purchased. Vendors which provide test and measuring equipment, standards, quality related service contracts, or subcontracted laboratory services (quality related items) shall be subject to the more rigorous controls below.

For the procurement of test and measuring equipment it is recognized that the environment in which the measurement system is placed may have a bearing on its performance. Therefore, the QA/QCC may substitute an acceptance testing plan to assure that the measurement system is able to meet specifications in the laboratory environment, in lieu of other supplier qualification activities.

4.2 Procurement of Quality Related Items

The quality of instruments, equipment, standards, reagents, solvents, other chemicals, gases, water, and laboratory containers used in analyses must be known so that their effect upon analytical results can be defined. Items purchased by an ITAS operating unit shall meet the requirements and specifications of client contracts or analytical methods, as detailed in laboratory-specific SOPs. At a minimum, all reagents shall meet the specifications established by the Committee on Analytical Reagents of the American

Chemical Society. If these are not available, a comparable or next best grade shall be used.

Quality specifications shall be included or referenced in the purchasing documents for the procurement of applicable items. The QA/QCC shall approve vendors or items purchased. This approval will be maintained by purchasing from a "QA Approved" list of vendors and items. If items which may affect laboratory quality are requested from non-preapproved vendors, QA approval must be obtained prior to placing the order.

When ordering such items, a system shall be put in place in each laboratory to assure the quality of the item received. Each laboratory shall assign individuals responsible for purchasing materials and controlling them in the laboratory. Duties of the responsible individuals include:

- Specifying in purchase orders or requisitions, suitable grades of materials (grade shall be defined by the QA/QCC or responsible manager)
- Verifying upon receipt that materials meet requirements and that, as applicable, material certificates are provided and maintained in the laboratory Quality/Operations records system
- Identifying and storing materials
- Verifying that material storage is properly maintained, and removing materials from use when shelf life has expired

4.2.1 Role of ITAS Division Purchasing

ITAS Division Purchasing supports the ITAS operations by:

- Maintaining contractual requirements of materials contracts
- Negotiating new contracts
- Identifying potential vendors and subcontractors
- Identifying vendors for unique or scarce materials

In order to enhance standardization of the product within the laboratory network, ITAS Division Purchasing shall pursue National Contracts for:

- Laboratory supplies of known quality and proven reliability
- Instrumentation
- Standards from traceable, certified sources

4.2.2 Procurement Procedures

The specifications for standards, chemical reagents, solvents, gases, water, and other items specified in approved analytical methods shall be met by the laboratory and

written in method SOPs. In addition, each laboratory must have the following items included in SOPs that cover:

- Checking the purity of standards, reagents, solvents, other chemicals, and water versus intended use
- Storage and expiration of standards, reagents, solvents, and other chemicals
- Requirements for laboratory containers (e.g. volumetric glassware, sample containers)
- Cleaning of glassware prior to use

Corrective actions for failure of an item to meet required specifications are:

- Review current supplies and eliminate from use
- Return to vendor
- Evaluate a new lot or alternate supplier

The Division Technical Director or the Division Director, QA/QC shall be immediately notified of any quality problems with national vendors.

4.2.3 *Radioactive Reference Materials*

ITAS shall procure only radioactive reference materials which are traceable to the National Institute of Standards and Technology (NIST). If the NIST traceability is not commercially accessible, the best available standard for that isotope shall be used. Certification of traceability shall be procured from the supplier. Documentation received with each standard shall include, at a minimum, the following information:

- Traceability to NIST or other certificate of analysis
- Radionuclide identification with activity and error
- Source identification or traceability number
- Date of assay
- Half-life of radionuclide(s)
- Mass and volume of standards
- Percent of impurities

Receipt, storage, use, control, and disposal of radioactive standards, as well as documentation of these activities, are described in operation-specific SOPs. Sources used to verify continuing calibration shall meet the requirements of this section.

4.3 *Procuring Services*

Subcontract Laboratory Services - A subcontract laboratory is defined, for the purposes of this QAMP, as a laboratory

external to the ITAS laboratory network. A subcontract laboratory will be used only in the event that an ITAS laboratory does not have the capability or capacity to perform the requested testing, or the customer so directs (in which case the customer then assumes responsibility for subcontractor performance). A subcontract laboratory will be used only after approval is obtained from the client and the quality of the laboratory is determined to be acceptable by ITAS QA/QC staff.

Once it is determined that a subcontract laboratory is required and approval is obtained from the client to use a laboratory external to the ITAS network, the QA/QCC must perform a quality systems audit of the selected subcontract laboratory. This audit must be approved by the Division Director, QA/QC and documented in both the Quality/Operations records at the laboratory and in division subcontract laboratory files. The procedure for the approval to use a subcontract laboratory is detailed in ITAS Division SOP No. IT-QC-0002.

4.4 Internal QC Requirements

4.4.1 Water

ASTM Type II grade or equivalent water at a minimum will be used in all metals, radiological, wet chemistry, and organic analyses. Type II water is obtained by the use of commercial ion-exchange deionizing units including an appropriate polishing unit. The resulting water has a maximum conductivity of 1.0 umho-cm at 25° C, minimum resistivity of 1.0 Mohm at 25° C, maximum total matter of 0.1 mg/L, a minimum color retention time of potassium permanganate of 60.0 minutes, and no detectable soluble silica. Conductivity and/or resistivity will be documented daily in a logbook or file. Maintenance documentation will be kept for both the deionizing units and the polishing unit.

For volatile analyses the water may be further purified by purging with an inert gas before use to remove traces of organic solvents.

Water monitoring procedures used by ITAS operating units are detailed in operation-specific SOPs.

4.4.2 Compressed Air and Gases

Ultra high purity compressed gases from preapproved vendors will be used when required for instrumentation. Compressed air and gasses must meet the requirements and specifications of the analytical methods performed. In-line filters will be used when appropriate to minimize contamination and moisture from the gases.

4.4.3 Glassware Preparation

Glassware preparation procedures instituted at ITAS operating units are designed to ensure that no contaminants are introduced during sample analysis. Procedures describing glassware preparation are detailed in operation-specific SOPs.

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5.0 DOCUMENT CONTROL AND RECORDS

ITAS has designed and implemented a system to control, distribute, and revise documents affecting quality. These documents are approved by the appropriate positions (see Table 2.3-1). Controlled documents are required to be reviewed and revised, if necessary, on a scheduled basis. The frequency of this review is dependent on regulations and client requirements, but it should occur at least annually. Interim changes to controlled documents may be instituted by the use of a Procedure Change Form (example shown in Figure 5.0-1). Procedure Change Forms require the approval of the laboratory QA/QCC, the Operating Unit Director, and the Division Director, QA/QC. Procedure Change Forms remain in effect until the next revision of the document. They are described in the ITAS System Procedure No. 8906-QAC-03, "Standard Operating Procedure for Preparation and Control of Procedures and Manuals". Controlled documents include but are not limited to this Operation-Specific QAMP, the ITAS QAMP, SOPs, and internally-generated QAPjPs and QAPPs.

5.1 Quality Assurance Management Plan (QAMP)

The ITAS QAMP provides ITAS Quality Assurance policy. It is applicable to and provides direction for all ITAS operations. The Plan discusses both the administrative and technical aspects of Quality Assurance and Quality Control. It is not however, intended that the Plan provide in-depth technical discussion. The Operation-Specific QAMP and SOPs supplement the ITAS QAMP to provide the details of implementation. The QAMP has precedence in policy matters over all other ITAS quality-related documents.

5.2 Operation-Specific QAMP (OS-QAMP)

The OS-QAMP supplements and enhances the ITAS-QAMP (Section 5.1). While the ITAS-QAMP serves as an over-view document and provides ITAS QA policy, the OS-QAMP further provides detailed technical discussions and information that is common to all ITAS operating units. An appendix for each operating unit supplements the OS-QAMP. Each appendix contains information that is specific to that operating unit only.

5.3 Manuals of Practice (MOP)

MOPs are developed to provide in-depth technical discussions of specific topics. For example, a MOP for the field collection, preservation, and shipment of samples to ITAS laboratories provides specific uniform

direction to IT Associates. The ITAS Chemical Hygiene Plan and Safety Manual are also considered MOPs. MOPs are usable across ITAS operations.

5.4 Standard Operating Procedures (SOPs)

SOPs are the foundation of the documented QA Program within ITAS. There are two levels of SOPs, division and operation-specific. Division SOPs specify methods that are common to all operating units and are standard across the network. Operation-specific SOPs detail procedures that pertain to a particular operating unit. All SOPs are written, detailed instructions describing specific laboratory operations and performance of routine laboratory tasks. They specify what is done, whose responsibility it is to perform tasks, and whose responsibility it is to verify their correctness. SOPs are sufficiently detailed to provide data of acceptable quality and integrity with a minimum loss of data due to out-of-control situations. They also provide for documentation to record the performance of all tasks and their results, and demonstrate the verification of the data each time the data are recorded, calculated, or transcribed. SOPs are written to address the major elements upon which analytical quality depends. ITAS has adopted a standard SOP format for use within the Division. The ITAS standard format for administrative SOPs is shown in Figure 5.4-1. The ITAS standard format for technical SOPs is shown in Figure 5.4-2.

5.5 Quality Assurance Project Plans (QAPjP)

Contractual and regulatory demands, or uniqueness of the scope of work of a project, may require the preparation and implementation of a project-specific QAPjP or QAPP. If a specific project requires a unique QA program, that program with full documentation must be provided to the ITAS operation for implementation. Full documentation will be provided in a QAPjP. The requirements of the project will take precedence over conventional ITAS QA practices for that work. The requirements of the project may not be less stringent than minimum ITAS QA/QC requirements unless requested by the client in writing or approved by the ITAS Division Director, QA/QC. Typical project requirements are as follows:

- The development and/or use of new or modified testing methods
- Special requirements for equipment calibration and maintenance
- Specific contract required detection limits (CRDLs)
- Defined data quality objectives (DQOs) such as accuracy and precision limits or the statistical treatment of data
- Additional or unique documentation or

records management requirements

5.6 Project and Quality/Operations Records Management

The ITAS QA Program has been developed to provide analytical results of known quality that meet client requirements. To demonstrate that quality has been achieved, each ITAS laboratory maintains a system of records management that includes documents which demonstrate the analytical performance of the laboratory. Laboratory records are identified as either Quality/Operations records or Project records.

All operating unit records, from time of sample receipt through reporting and disposal, shall be available and stored in a manner that safeguards their integrity from tampering, physical damage, and/or loss. For ITAS, this will be separate files in file cabinets in a 24 hour per day secure area at a minimum. Any documentation that directly bears on the reported results must be retrievable if requested by the client or legally compelled by an authorized regulatory agency or court of law. This includes operational and project-specific data. Data may be stored in "real-time" as it is produced, or filed in a manner to allow prompt retrieval and assembly into a complete project file. Operation-specific SOPs shall describe how the complete set of documentation is compiled, including the flow of data forms, locations, responsibilities, and checks on the records management system implemented.

5.6.1 Project Records

Project records are documents which are specific to a project or a group of samples within an ongoing project, such as chain-of-custody and raw analytical data. Project records are stored separately in project files. Each project file shall be indexed, properly labeled, and current.

ITAS will maintain records associated with specific projects as nonpermanent records for the following time periods after completion of a project:

- Analysis performed as part of site mitigation activities - 10 years
- Records associated with facilities governed by the Resource Conservation Recovery Act (RCRA) - 5 years after closure if the analysis was performed prior to closure or for the 30-year monitoring period following closure if the analysis was performed for the purpose of closure monitoring
- Conventional analysis - 7 years

If a special contractual requirement, project requirement, or government regulation requires that records be maintained for a longer period of time, project files will be

kept as required. For projects that must be kept beyond the periods stated above, the project index shall be marked to indicate the required retention period.

Prior to scheduled record destruction, records shall be reviewed to determine if the holding period should be extended.

5.6.2 Quality/Operations Records

Quality/Operations records are permanent (lifetime) documents which demonstrate overall laboratory operation, such as instrument logbooks, calibration data, and control charts. These records will directly affect the data for a specific project, but in general their applicability is not limited to one project. Quality/Operations files must be indexed, properly labeled, and current.

5.6.3 Record Validation

When records are transferred to a records storage area, they shall be verified by comparing the contents of the container against an inventory sheet listing the contents of the container. If there are any discrepancies, the container and inventory sheet shall be returned to the Associate submitting the records for resolution. When the document container and the inventory sheet are found to be acceptable, the responsible records custodian shall indicate approval by signature and date on the inventory sheet. Records shall be considered valid when the inventory sheet is signed by the custodian[1].

[1]NQA-1, Supplement 17S-1, Section 2.3.

6.0 USE OF COMPUTER HARDWARE AND SOFTWARE

The purpose of defining controls for computer hardware and software is to protect the integrity of computer-resident data in the laboratory. SOPs are in place in each location so that computer resident data are accurate and defensible. The following references shall be used as guidance for implementation of this system of controls:

- Good Automated Laboratory Practices, US Environmental Protection Agency, (draft), December 28, 1990
- Quality Management and Quality Assurance Standards ISO 9000, Part 3: "Guidelines for the Application of ISO 9001 to the Development, Supply and Maintenance of Software"
- ASTM Method E3140-1 (draft): "New Standard Guide for Laboratory Information Management Systems (LIMS)"
- ANSI N413: "Guidelines for the Documentation of Digital Computer Programs"

6.1 Use of Hardware

Computer equipment used in the generation, measurement, or assessment of client data shall be appropriately designed, be of adequate capacity to function according to specifications, and shall be suitably located for operation, inspection, cleaning, and maintenance. There shall be a written description of the computer system(s) hardware. The computer shall be installed in accordance with manufacturer's recommendations and undergo validation which demonstrates the computer equipment correctly performs its stated capabilities and functions. Changes to computer hardware shall be made only after review and approval of the LIMS Manager and Laboratory Director.

Computer hardware shall be inspected, cleaned, and maintained on a regular basis at a minimum of annually. Each laboratory shall:

- Have SOPs for the maintenance and security of hardware
- Designate an Associate (usually the LIMS Manager) to be responsible for system performance

6.2 Security

Each operating unit shall have procedures in place which secure computer hardware and software systems if that system:

- Contains confidential information that requires protection from unauthorized disclosure
- Contains data in which the integrity must be protected against unintentional error or intentional fraud

- Is used to acquire, process, or report data

When the computer system(s) contains data that must be secured, each operating unit shall ensure the system is physically secured. Physical and functional access to the system is limited to authorized Associates and introduction of unauthorized external programs or software is prohibited.

The LIMs Manager, or designee, is responsible for maintaining a list of secure, licensed software, and software not on that list is considered unauthorized.

All original software must be stored in a locked, secure area.

6.3 Use of Software

If computer software is used to acquire, process, or report client data, it is necessary to demonstrate that the software correctly performs its intended function. The following definitions are important to this discussion:

- Validation - establishing documented evidence which provides a high degree of assurance that a specific process will consistently produce a product meeting its predetermined specifications and quality attributes. This process demonstrates that the mathematical or statistical model embodied in the computer program is an acceptable representation of the process or system for which it is intended and meets all specified requirements.

- Verification - the process of checking the accuracy of manually or automatically (electronically) entered information.

In general, software is verified by comparing its performance against known results. Verification may be done in several ways (see Sections 6.3.1 and 6.3.2). Each operating unit shall have a software SOP(s) describing the following:

- Software verification and validation
- Data entry and verification
- Changing data
- Data analysis, processing, storage and retrieval
- Backup and recovery of data and software
- Electronic reporting of data
- Definition and storage times for data and software

6.3.1 Industry Standard Software

Industry standard programs are defined as programs which are widely used throughout the profession. These standard programs are brought into ITAS and used without modification. If the program has been prepared external to IT, independent validation is not required. However, the

program must be verified prior to first use on an ITAS system. To verify the software, example problems must be processed to demonstrate that the program is fully operational. Example problems must fully test the utilized capabilities of the software.

6.3.2 ITAS Developed Software

For programs developed within ITAS and externally prepared programs which are modified by ITAS, complete validation and verification must be performed. Validation must be performed in accordance with an approved reference (Section 6.0) and IT Standard Quality Practice, ITC0010 "Software Development and Usage". The verification process is dependent upon the function of the software and should include:

- For software which only performs numerical manipulation, sample sets of numbers for which results are known should be processed and compared. In this case, known results are usually generated by performing hand calculations using the same equations and procedures as the software. Verification of the software must test the software production of the intended results. Problems must test both the theory, or basis for computation, and the ability of the software to store and manage data.

- Software which performs as part of instrument operation should be verified by processing reference materials through the instrument system. Processed instrument response shall be compared against the standards used. Verification shall be performed annually, at a minimum.

6.3.3 Control of Software Changes

Changes to software shall be controlled. Detailed software control procedures must be available in each ITAS laboratory. Standard forms are used to document and track changes. An ITAS Associate in each laboratory must be assigned to maintain software control, usually the LIMS Manager or programmer. Whenever a program is changed, reverification is necessary. If the software has had features added, previous test problems should be rerun to demonstrate their function has not been affected. New test problems should be processed as discussed above to verify added performance. If software revision changes the basic operation of the program, complete revalidation and reverification of the program is required. All changes must be completely documented.

6.3.4 Software Review and Reverification

Spreadsheets and unprotected software shall be reverified on an annual basis at a minimum. The test problems used to provide initial verification shall be reprocessed and the results compared to demonstrate that performance of the software is unchanged. If software performance has changed, the effect of the change upon intended function and usage

since last verification shall be assessed. "Effect" must be determined on a case-by-case basis for the scope and impact of incorrectly reported results. If necessary, the data shall be reprocessed and recipients of affected data reports notified. Software programs must be validated upon creation or change and verified annually.

6.3.5 Software Validation and Verification Documentation

Software validation and verification shall be documented by the Associate performing the work, by signing and dating in indelible ink the computer output and supporting calculations. When test problems are used, the input shall be marked to indicate correct usage and the output checked to indicate acceptable comparison. If reference materials are used as the basis for validating and verifying instrument software, the "true" values or certificates for the materials shall be included with the output to demonstrate performance. The verification documentation must be reviewed and approved by the Associate's immediate supervisor, the LIMS Manager or computer programmer, and QA/QCC.

All software validations and all software verifications, whether for initial or subsequent reverification, shall be maintained in the Quality/Operations records management system. A historical file shall be maintained for each program. The file shall include the basis for the verification, such as the test problems or hand calculations, results of the software performance, the results of subsequent reverifications, applicable program code, user manuals, technical documentation and a copy of the program.

6.4 Computer Viruses

ITAS operating units shall employ the use of anti-virus software to detect and remove viruses from secure computer hardware. Any suspicion of a software virus must be immediately reported to the LIMS Manager, the Division Technical Director, and the Division Director, QA/QC.

7.0 WORK PROCESSES AND OPERATIONS

Much of the environmental project activity is planned and designed external to the laboratory or field operation and presented in the form of a contract, work plan, or QAPjP. Laboratory and field activities are planned, implemented, and assessed to meet client requirements according to approved procedures and methodologies. Many QA systems have been put in place to document the implementation of planned activities. The planning and design of operational systems to accomplish documented implementation are detailed in operation-specific SOPs. The entire process is assessed on a regular basis for conformance to prescribed requirements.

7.1 Standard Operating Procedures

SOPs are in place in all ITAS operating units for analytical and administrative procedures from the receipt of samples in the laboratory through analysis, reporting, and subsequent sample disposal. This includes auxiliary functions as well, such as training, QA/QC, and Health and Safety procedures. The ITAS standard SOP formats are shown in Figure 5.4-1 (administrative) and Figure 5.4-2 (technical). A list of ITAS operating unit SOPs is given in Appendix Section 3.

ITAS operations prepare and maintain, in addition to the Operation-Specific QAMP, a Standard Operating Procedure Manual. The requirements of this OS QAMP for activities such as calibration, field procedures, material procurement and control, preventive maintenance, training, and QC sample analysis shall be incorporated into the SOPs as appropriate.

7.2 Analytical Methods

Whenever possible, ITAS operations utilize industry and regulatory agency recognized analytical methods from source documents published by agencies such as the U.S. Environmental Protection Agency (USEPA), American Society for Testing and Materials (ASTM), and the National Institute for Occupational Safety and Health (NIOSH). Analytical methods used by each ITAS operation are listed in Appendix Section 4.

7.3 Detection Limits

All chemical analytical methodologies have an associated detection limit below which an analyte present in the sample cannot be accurately determined. A detection limit value may be reported in one of three ways:

- As a less than (<) value
- As not detected (ND)

- As an undetected (U) value

In all cases, the detection limit will also be reported for reference.

Detection limits indicated in methods are highly matrix dependent and are provided for guidance. Depending upon the exact sample composition, stated detection limits may not always be achievable.

The method detection limit (MDL) is defined by the USEPA as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. ITAS uses USEPA Procedure CFR 40 Part 36, Appendix B to determine detection limits. MDLs are determined annually.

For radiochemistry, whether the net result is negative, zero, or positive, the actual calculated result is reported with its associated propagated uncertainty. The detection limit is affected by many factors, such as the length of count, chemical yield, half-life, background of the instrument, counting efficiency, and the matrix interference. The minimum detection limit for radiochemical analyses is defined as the smallest activity of material that yields a net count above background with a 95 percent probability and no greater than a 5 percent probability of calling a blank a true signal. Detection limit calculations, frequency determined, and exact procedures for specific analytical methods are documented in operation-specific SOPs.

Method Detection Limits (MDLs), Practical Quantitation Limits (PQLs), and CRDLs (when applicable) are listed in Appendix Section 5.

7.4 Variance from Stated Methods

Work processes will be performed in accordance with SOPs derived from the methods referenced unless specific project requirements or needs dictate adoption of an alternate method or modification of the cited methods. For example, GC/MS procedures may be "USEPA CLP-modified" if specified by a client.

For some matrices and analytes, ITAS has developed in-house SOPs based on regulatory methods which may include modifications to improve reproducibility and/or accuracy. If an operation is performed in an alternate manner, the method shall be documented in the project records.

7.5 Assessment of Work Processes

All work processes or operations are subject to assessment as described in Section 9.4.

8.0 DATA COLLECTION AND PRODUCTION OPERATIONS

Laboratory analyses are designed to produce data that are representative of existing conditions present at the time the sample was obtained. The data collection design includes field sampling events, sample handling and custody, analytical operations, data verification, techniques to assess limitations of data use, and data reporting requirements.

In order to provide a sample that most accurately represents the test matrix, field sample collection personnel must abide by the sample collection guidelines and procedures established by involved regulatory agencies. A significant part of the efforts of regulatory agencies include the use of "approved" sample containers, chemical and physical preservation techniques, and observance of specified holding times. Although at times the sampling may be performed by non-ITAS Associates, the importance of sampling and transportation of the sample to the laboratory is understood and must be considered during data validation. Figure 8.0-1 is a flow chart showing the data collection process. The steps presented are described in detail in the following sections.

8.1 Field Collection and Shipment

In order for data to be representative of existing conditions present at the time the sample was collected, it is imperative that all samples be collected and preserved according to the appropriate analytical method specified in the QAPjP or QAPP (if one exists). Sampling requirements must be communicated to the sampling team prior to field collection.

Field personnel are responsible for labeling each individual sample collected with the following information:

- Project number
- Unique sample number
- Sample location (including as appropriate: borehole and depth or grid coordinates)
- Sampling date and time
- Person(s) obtaining the sample
- Sample preservation
- Analysis required

An overriding consideration for the resulting analytical data is the ability to demonstrate that the samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Evidence of collection, shipment, laboratory receipt, laboratory custody, and disposal must be documented

to accomplish this. Figure 8.1-1 shows the Analysis Request/Chain-of-Custody (AR/COC) form that is used by the ITAS laboratory network.

Field personnel are responsible for initiating the AR/COC form. In addition, they are responsible for uniquely identifying and labeling samples, providing proper sample preservation, and packaging samples to preclude breakage during shipment.

The prompt shipment of samples to the laboratory is required to ensure that required holding times are met. Samples should be shipped by an overnight carrier, be hand-delivered, or transported in a manner that assures prompt delivery to the laboratory.

Some sites require an extensive radioactive screening process before a sample may be shipped. In these cases, it is imperative for the project manager to maintain good communications with the client to assure proper staffing of the laboratory in response to a decreased holding time.

Radioactive samples that are shipped to ITAS operations must be screened and found not to contain radioactivity that exceeds the level stated in the operation license of the laboratory. Samples received by an ITAS operation that contain radioactivity exceeding their license limit will immediately be returned to the project site.

8.2 Sample Containers, Shipping Containers, Preservatives, and Holding Times

8.2.1 Sample Containers

A sample container is defined as the sealed enclosure, usually made of borosilicate glass or plastic, that the sample is collected in and stored in until analysis. All sample bottles provided by ITAS operations for environmental sampling are purchased new and are certified precleaned following appropriate USEPA procedures by the manufacturer. The bottles to be supplied for inorganic analyses are listed in Table 8.2-1. The bottles to be supplied for organic analyses are listed in Table 8.2-2. Radiological sample bottles are listed in Table 8.2-3. All documentation certifying bottle cleanliness must be maintained in the operation's Quality/Operation files.

8.2.2 Shipping Containers

Shipping containers are defined as the sealed enclosure in which the sample containers are stored during shipment from the sample collection site to the analytical laboratory. Shipping containers must be of sufficient number and size to accommodate the samples in an upright condition. Shipping containers must also meet all Department of Transportation (DOT),

International Air Transportation Association (IATA), International Civil Aviation Organization (ICAO), United Nations (UN), USEPA, and Nuclear Regulatory Commission (NRC) requirements for the shipment of environmental and/or radioactive samples.

Packaged samples must be shipped to the analytical laboratory in a safe manner that preserves the integrity of the samples. The most common method of sample shipment employs coolers or ice chests that are sealed with custody tape and shipping tape. These coolers must be durable and resistant to crushing during shipment. All coolers must be well maintained and cleaned to prevent cross contamination of the samples. It is the ultimate responsibility of the person collecting and packaging the sample for shipment to ensure that the shipping containers are clean and functional. To help prevent sample breakage during shipment, additional consideration must be given to providing shock absorbency to all samples packaged inside the shipping container. Use of bubble-wrap around each sample container is the best way to provide this protection, followed by foam packing materials and vermiculite which are also commonly used.

8.2.3 Sample Preservatives

Most analytes have a finite holding time in a given sample matrix. Sample preservation is the chemical or physical means by which samples are treated during and/or following sample collection to aid in the stability of the analytes of interest for a given sample matrix. The preservation of samples at the time of sample collection will follow the requirements of the analytical methods used. This preservation includes the addition of reagents to deter chemical degradation and the maintenance of refrigeration during transit and ultimate storage in the laboratory. The required preservatives for the analysis to be performed on each matrix are included in Table 8.2-1 for all inorganic analyses and Table 8.2-2 for all organic analyses. Radiological sample preservatives are listed in Table 8.2-3.

8.2.4 Sample Holding Times

Holding time is defined as the maximum allowable time between sample collection (or laboratory receipt for CLP) and extraction or analysis. ITAS has developed a commitment to meeting sample holding times that extends throughout each ITAS operating unit. Each operation has a system in place to ensure that holding times are monitored by each group within the operating unit. It is the responsibility of each ITAS Associate processing the sample to assure that holding times are met. ITAS laboratories are responsible for meeting all

holding times for samples received within 24 hours of collection.

The holding times for inorganic analyses are listed in Table 8.2-1. The holding times for organic analyses are listed in Table 8.2-2. Radiological sample holding times are listed in Table 8.2-3.

8.2.5 Turnaround-Time (TAT)

TAT is defined as the maximum number of days from sample collection to the date Certificates of Analysis or other deliverables are received by the client. Sample collection personnel must be aware of the holding time and TAT requirements so that they can determine the best method of sample transport and can communicate that information to the laboratory Project Manager.

8.3 Sample Receipt and Initiation of Testing Program

Each ITAS laboratory has a SOP describing this program in detail. The following sections describe the general program followed by all ITAS operating units.

8.3.1 Sample Receipt

Samples shall be received and logged in at ITAS operations by a designated sample custodian or properly trained designee back-up. Upon sample receipt, the sample custodian shall, as appropriate:

- Wear appropriate personal protective equipment (PPE). At a minimum, this consists of gloves, a lab coat, safety glasses, and in some cases a respirator
- Examine the shipping containers to verify that the custody tape is intact
- Examine all sample containers and determine if the temperature required by the requested testing program (normally 4°C ± 2°C) has been maintained during shipment. Document shipping container temperature on the AR/COC
- Examine all sample containers for damage
- Compare samples received against those listed on the AR/COC
- Verify that sample holding times have not been exceeded
- Examine all sample paperwork for correctness and completeness
- Determine sample pH (if required for the scheduled analysis) and record on the AR/COC
- Sign and date the AR/COC immediately (only after shipment is accepted) and attach the waybill
- Note any problems associated with the samples on the AR/COC, immediately initiate a Condition Upon Receipt Variance Report (CUR), and notify the Project Manager who in turn notifies the client
- Attach appropriate laboratory sample container labels with laboratory ID, test,

- Place the samples in proper laboratory storage

8.3.2 Sample Log-in

Sample log-in activities at ITAS operating units are fully documented in operation-specific SOPs. The following is a general description of the log-in process:

- Enter the samples in the laboratory sample log-in book, and/or the sample management computer system (LIMS) which contains the following information at a minimum:
  - Project identification number
  - Sample numbers (both client and laboratory)
  - Type of samples
  - Required tests
  - Date received in laboratory
- Notify the Project Manager and appropriate Group Leader(s) of sample arrival
- Place the completed AR/COCs, waybills, and any additional documentation in the project file

8.3.3 Sample Storage

The primary considerations for sample storage are:

- Maintenance at prescribed temperature, if required, which is typically 4°C ± 2°C
- Processing samples within the prescribed holding time for the parameters of interest
- Maintenance of sample integrity through adequate protection from contamination from outside sources or from cross-contamination of samples. Low-level and high-level samples, when known, must be stored separately. When applicable, samples and standards must be stored in separate refrigerators or freezers.

The requirements listed on Tables 8.2-1, 8.2-2, and 8.2-3 for temperatures and holding times shall be used. Placing of samples in the proper storage environment is the responsibility of sample control personnel who shall notify the Operations Manager and Group Leaders if there are any samples which must be analyzed immediately because of holding time requirements.

8.3.4 Internal Sample Chain-of-Custody

Internal sample custody is tracked and documented in ITAS laboratories as described in operation-specific SOPs. The sample custody documentation shall include, but is not limited to the following:

- Signatures for relinquishing and receiving samples or sample extracts
- Listing of all procedures (sample preparation and analysis) performed on the sample and sample extract

- Laboratory and project identification
- Sample matrix
- Laboratory sample numbers

8.3.5 Sample Disposal and Return Chain-of-Custody

After the requested analyses on the samples have been completed, any remaining portions of the samples will be maintained by the sample custodian until the samples are disposed of or returned to the client. The disposal of each sample is recorded on the client's Chain-of-Custody (COC) form or referenced in the project file. Sample disposal procedures and documentation are described in operation-specific SOPs.

For NRC or state licensed laboratories, a real time inventory of all radioactive isotopes contained in the laboratory (including radioactive samples), as required by the NRC or state, is maintained by the Radiation Safety Officer (RSO). If the quantities of radioactive materials in-house approach the limits of the laboratory NRC license, appropriate action will be taken to ensure the license is not exceeded. This may involve returning samples to clients immediately.

The original copy of the client's COC form will be maintained in the appropriate laboratory project file unless the entire sample is physically transferred off-site. In that case, the original COC record will be signed off as relinquished by the sample custodian or designee and will accompany the sample in shipment. A copy of the completed COC form will be retained in the laboratory project file. In the case where an aliquot of a sample is shipped from the laboratory, a new COC will be generated by the laboratory and shipped with the sample aliquot and the original COC will be retained in the project file.

8.4 Calibration Procedures and Criteria

8.4.1 Calibration System

All equipment and instruments used at ITAS operations for quantitative measurements are controlled by a formal calibration program. Two types of calibration are discussed in this section. These are operational and periodic calibrations. Operational and periodic calibration procedures are described in operation-specific SOPs. At a minimum, these procedures shall include:

- Equipment to be calibrated
- Reference standards used for calibration
- Calibration technique and sequential actions
- Acceptable performance tolerances
- Frequency of calibration

- Calibration documentation requirements

Whenever possible, recognized procedures, such as those published by ASTM or the USEPA, or procedures provided by manufacturers shall be adopted. If established procedures are not available, a procedure shall be developed considering the type of equipment, stability characteristics of the equipment, required accuracy, and the effect of operational error on the quantities measured.

8.4.2 Reference Standards

Two types of reference standards are used within ITAS operations for calibration: physical reference standards and chemical reference standards.

8.4.2.1 Physical Reference Standards

Physical reference standards include weights for calibrating balances and certified thermometers for calibrating working thermometers. These are generally associated with periodic calibrations. Whenever possible, physical reference standards shall have known relationships to nationally recognized standards such as the National Institute of Standards Technology (NIST), formerly the National Bureau of Standards (NBS). If national standards do not exist, the basis for the reference standard shall be documented.

Physical reference standards shall be used only for calibration procedures and shall be stored separately from equipment used for analysis.

8.4.2.2 Chemical Reference Standards

These standards are generally associated with operational calibration. Chemical reference standards include Standard Reference Materials (SRMs) provided by NIST, the USEPA, or other recognized standards agency. This may include vendor-certified materials traceable to NIST or USEPA SRMs. Radioactive reference materials are discussed in Section 4.2.3.

8.4.2.3 Standard Verification

Standard verification is performed at all ITAS laboratories by comparison of a standard with a SRM or second-source standard. For chemical analyses, ITAS operating units shall use purchased standard mixes, when possible, from two different sources. The response factors of the two shall be compared. Neat standards shall be used only when standard mixes are not available. If only one standard source is available, the laboratory shall have two different analysts prepare the stock solution and dilutions of the stock solution. The laboratory shall then compare the response factors of these two separately prepared standards. In the rare cases, such as dioxin standards where costs are significant, new

standards shall be compared to previous standards for verification. Operation-specific SOPs shall define the specific requirements and process of standard preparation and verification.

8.4.3 Operational Calibration

Operational calibration is routinely performed as part of instrument usage, such as the development of a standard calibration curve. Operational calibration is generally performed for instrument systems.

A summary of the various operational calibrations performed at ITAS operations is shown in Tables 8.4-1 for inorganic method calibrations, and 8.4-2 for organic method calibrations.

8.4.4 Periodic Calibration

Periodic calibration is performed at prescribed intervals. In general, equipment which can be calibrated periodically is a distinct, singular purpose unit and is relatively stable in performance. ITAS operations perform this type of calibration on balances, micropipettors, and thermometers.

Each ITAS operating unit has SOPs in place for the calibration of equipment requiring periodic calibration. Periodic equipment calibrations employed at ITAS operations are listed along with their respective calibration criteria in Table 8.4-3.

8.4.5 Calibration Failure

Equipment that fails calibration or becomes inoperable during use shall be removed from service and segregated to prevent inadvertent use, or shall be tagged to indicate it is out of calibration. Such equipment shall be repaired and recalibrated before reuse.

Recalibration may occur more frequently than scheduled. At any time, if equipment calibration becomes suspect, it shall undergo a calibration check to determine whether the current calibration is still acceptable or if recalibration is required.

8.4.6 Calibration Records

Calibration records shall be prepared for each piece of equipment subject to calibration and shall be maintained according to operation-specific SOPs.

All calibration records (operational and periodic) directly affect data and may not be limited to one project. These records shall be stored in the operating unit Quality/Operations files unless it is required for a project that they be stored in the project file.

8.5 QC Sample Analysis

QC samples are routinely added to the

normal laboratory sample stream to demonstrate that the laboratory is operating within prescribed requirements for accuracy and precision. Statistical evaluation of QC sample data is discussed in Section 9.2. It is mandatory that all types of QC samples be handled and treated in the field and in all areas of the laboratory in the same manner as actual client samples.

QC levels and types of QC samples are described in the following sections. Laboratory QC samples are also listed per type of analysis in Table 8.5-1 for inorganic analyses and Table 8.5-2 for organic analyses. These tables also list the required frequency, acceptance criteria, and corrective actions. Radiochemistry QC samples are listed in Appendix Section 8 where applicable.

ITAS requires a minimum QC sample analysis frequency of 15%. When QC sample analysis requirements are not specified by a method, the following minimum will be used:

- Method blank
- Laboratory Control Sample
- Matrix Spike sample or Duplicate sample

8.5.1 QC Levels

Following are definitions of the ITAS QC levels:

Level I: ITAS Standard Practice. Use available analytical procedures. Fifteen percent QC samples (blank/spike/duplicate or duplicate spike) for every 20 samples of a given matrix. QC samples may not be performed for a specific project, but as part of compiled sets of samples. QC data are not reported with the analytical results.

Level II: ITAS Standard Practice/Project Specific. Use available analytical procedures. Fifteen percent QC samples minimum (blank/spike/duplicate or duplicate spike) for every 20 samples of a given matrix. QC samples are client or project specific. QC summary report is included with the analytical results. No raw data are included.

Level III: CLP or Equivalent. Use referenced regulatory procedures and/or established/verified procedures using confirmatory techniques. Method blank plus two QC samples minimum per each matrix. QC summary report is supplied with supporting data. Where applicable, this is the USEPA CLP package.

Level IV: Project Specific. QC requirements are defined in a QAPjP, Work Plan, Contract or other specific plan or procedure. Project documentation must be submitted to the laboratory prior to sample submittal.

8.5.2 Field QC Samples

When field QC sample collection and analysis are required for a project, it is the responsibility of the project sampling supervisor to ensure that this sampling is performed correctly and at the project-required frequencies. Field QC samples may or may not be identified as such to the laboratory and will undergo sample preparation and analysis procedures identical to the actual field samples. Field QC sample results are reported in the Certificate of Analysis or other project-required deliverable. The results are stored in the project file with which they are associated.

Field QC sample types, applicability to organic and inorganic analyses, precision and accuracy applications, and persons they are introduced by are summarized in Table 8.5-3. The following sections provide descriptions of field QC samples.

8.5.2.1 Trip (Travel) Blank (TB)

Volatile organics samples are susceptible to contamination by diffusion of organic contaminants through the Teflon-lined, silicone, rubber septum of the sample vial. Therefore, Trip Blanks, also referred to as Travel Blanks, shall be analyzed to monitor for possible sample contamination during shipment. TBs will be prepared by filling two VOA vials (40 ml) with organic-free water and shipping the blanks with the field kit. TBs accompany the sample bottles during collection and shipment to the laboratory and are stored with the samples.

8.5.2.2 Field Blank (FB)

A FB is a volume of water (or soil) that is provided by the sample collectors to demonstrate the absence of contamination during sampling. Deionized, distilled laboratory water, or previously-prepared solid materials (i.e. lab sand) is placed into sample containers by the sample collection crew, packaged, and shipped with the other field samples.

8.5.2.3 Rinsate Blank

A rinsate blank is a volume of rinse solution (deionized, distilled lab water or organic solvent) used to rinse a sampling tool which contacts multiple samples. The rinse solution is collected after the tool has collected a sample and has been cleaned, to demonstrate that there is no residual contamination remaining on the tool to carry over into the next sample.

8.5.2.4 Field Duplicate (FD)

A FD sample is a duplicate sample which has been introduced as a separate sample by the sample collection personnel. Results of FD samples provide a measure of field precision.

8.5.2.5 Field Matrix Spike Analyses

A field MS sample is created by spiking target analytes into a portion of a sample in the field at the point of sample acquisition. This sample provides information on the target analyte stability after collection and during transport and storage.

8.5.2.6 Collocated Samples

Collocated samples are independent samples collected in such a manner that they are equally representative of the variable(s) of interest at a given point in space and time. Examples of collocated samples include: samples from two air quality analyzers sampling from a common sample manifold or two water samples collected at essentially the same time and from the same point in a lake.

Collocated samples processed and analyzed by the same organization provide intralaboratory precision information for the entire measurement system including sample acquisition, handling, shipping, storage, preparation, and analysis. Both samples can be carried through the steps in the measurement process together to provide an estimate of short-term precision for the entire measurement system. Likewise, the two samples, if separated and processed at different times or by different people, and/or analyzed using different instruments, provide an estimate of long-term precision of the entire measurement system.

Collocated samples processed and analyzed by different organizations provide interlaboratory precision information for the entire measurement system.

8.5.2.7 Replicated Sample Analyses

A replicated sample is a sample that has been divided into two or more portions at some step in the measurement process. Each portion is then carried through the remaining steps in the measurement process.

8.5.2.8 Split Sample

A split sample is a sample divided into two portions. One portion is sent to a different organization or laboratory and subjected to the same environmental conditions and steps in the measurement process as the portion retained.

A split sample can be divided into portions at different points in the sampling and analysis process to obtain precision information on the various components of the measurement system. For example, a field split sample provides precision information about all steps after sample acquisition including the effects of storage, shipment, analysis, and data processing; whereas, information on the intra- and

interlaboratory precision of sample preparation and analysis steps of the measurement system is provided by samples split once they are received in the laboratory.

8.5.3 Laboratory QC Samples

Laboratory QC samples, when successfully analyzed, demonstrate that the functions which are under laboratory control are within acceptable limits. Any laboratory QC sample results that are outside of acceptable limits must be documented on a nonconformance memo. Laboratory QC sample types, applicability to organic and inorganic analyses, precision and accuracy applications, and persons they are introduced by are summarized in Table 8.5-5. In addition, Tables 8.5.1 (inorganic) and 8.5-2 (organic) list laboratory QC samples, acceptance criteria, and corrective actions per analytical method. The following sections provide descriptions of laboratory QC samples.

8.5.3.1 Method Blank (MB)

A MB is a volume of deionized, distilled laboratory water for water samples, or a purified solid matrix for soil/sediment samples carried through the entire analytical procedure. The volume or weight of the blank must be approximately equal to the sample volume or sample weight processed. A MB shall be performed with each group of samples. Analysis of the blank verifies that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware are known and minimized. Optimally, a MB should contain no greater than the reporting limit for the parameter. Results of MB analyses shall be maintained with or referenced to the corresponding analytical data in the project file.

8.5.3.2 Reagent Blank (RB)

A reagent blank is composed of the materials which will be added to client samples during preparation, and analyzed for specific parameters. It is analyzed to verify that no laboratory contaminants are present at levels which would affect sample results. RBs must be successfully analyzed prior to sample analysis. Records of associated solvent lots and column absorbent test results are stored in Quality/Operation files.

8.5.3.3 Duplicate Sample Analyses

Duplicate analyses are performed to evaluate the precision of an analysis. Results of the duplicate analyses are used to determine relative percent difference. Criteria for evaluating duplicate sample results are provided in Section 9.2.

8.5.3.4 Continuing Calibration Standard (CCS)

Because standards and calibration curves

are subject to change and can vary from day to day, a midpoint standard or check standard should be analyzed during each time period required by the analytical method. Analysis of this standard is necessary to verify the standard curve and may serve in some cases as sufficient for calibration.

8.5.3.5 Surrogate Standard (SS)

A SS determination should be performed on all samples and blanks for methods requiring surrogate usage. Surrogates should be similar to the target analytes in chemical composition and behavior in the analytical process, but not normally found in environmental samples. All samples and blanks are fortified with surrogate spiking compounds before purging or extraction to monitor preparation and analysis of samples.

8.5.3.6 Laboratory Matrix Spike (MS)

To evaluate the effect of the sample matrix on analytical methodology, a separate aliquot sample should be spiked with the analyte(s) of interest and analyzed with the sample. The percent recovery for the respective compound will then be calculated. If the percent recovery falls outside established QC limits, the data and other associated QC sample results should be evaluated and the sample may require reanalysis if criteria are not met. This type of MS does not necessarily reflect the behavior of the field-collected target analyte, especially if the target analyte is not stable during shipping or storage.

8.5.3.7 Laboratory Matrix Spike Duplicate (MSD)

Similar in concept to the MS sample, a MSD is a separate aliquot sample that is spiked with the analyte(s) of interest and analyzed with the associated sample and sample matrix spike. If the percent recovery falls outside established QC limits, the data and other QC sample results should be evaluated and the sample may require reanalysis if criteria are not met. The comparison of the recoveries of the spiked compounds in the MS and MSD samples is made to determine the relative percent difference (RPD) between the MS/MSD samples.

8.5.3.8 Laboratory Control Sample (LCS), Blank Spike (BS), or QC Check Standard

A LCS is a blank sample spiked with the parameters of interest or is a matrix of known parameter concentrations that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of spiked materials demonstrates that the laboratory techniques for this method are in control. Where required, this sample is analyzed in conjunction with MS/MSD samples. If the

MS/MSD pair shows poor recoveries due to interferences or matrix effects, yet the LCS is acceptable, this is strong evidence that the method has been performed correctly by the laboratory for these samples, but the sample matrix has affected the results. Results of LCS analyses must be cross-referenced with the corresponding MS/MSD and sample analytical data in the project file. LCSs are control charted to graphically demonstrate laboratory control or monitored through use of control tables. LCSs are described in the ITAS Division SOP No. ITAS-IT-QC-0004, "Use and Purpose of Laboratory Control Samples."

8.5.3.9 Analytical Spike (AS)

An AS sample is created by spiking target analytes into a prepared portion of a sample just prior to analysis. It provides information on matrix effects encountered during analysis such as suppression or enhancement of instrument signal levels. It is most often encountered with elemental analyses involving the various forms of atomic spectroscopy and is often referred to as the "method of standard additions" (MSA).

8.5.3.10 Internal Standard Spike (IS)

An IS is an analyte which has the same characteristics as the surrogate, but is added to a sample just prior to analysis. It provides a short-term indication of instrument performance, but it may also be an integral part of the analytical method in a non-quality control sense, e.g., to normalize data for quantitation purposes.

8.5.4 Matrix QC Samples

Matrix QC samples include MS, MSD, and duplicate samples which are discussed in Section 8.5.3.

8.5.5 Radiological QC Samples

Radiological QC samples are listed in Section 8 of the OS QAMP appendices when applicable. The following is a discussion of QC samples that are specific only to radiochemical analyses.

8.5.5.1 Yield Monitors

Yield monitors are added to the actual samples. There are two types of yield monitors: tracers and carriers. A tracer is a radioisotope usually of the same element, and usually having the same mode of decay as the analyte, that is added to the sample to monitor recovery. A carrier is a non-radioactive solution added to assist in isolating the specific isotope of an element. When standardized, the carrier can also provide recovery information gravimetrically.

8.5.6 Performance Evaluation Samples (PEs)

PE samples may be blind or double-blind

samples are discussed in detail in section 9.4.1.

8.6 Data Reduction, Verification, and Reporting

Data review procedures, ideally defined as a set of computerized and manual checks applied at various appropriate levels of the measurement process, will be clearly defined for all measurement systems in operation-specific SOPs. Responsibilities for data and report review are defined in the ITAS Division SOP No. ITAS-IT-QC-0003, "ITAS Data and Report Review Responsibilities". Data review begins with the reduction (processing) of data and continues through verification of the data and the reporting of analytical results. Calculations are checked from the raw data to final value prior to reporting results for each group of samples. Data reduction can be performed by the analyst who obtained the data or by another analyst. Data verification starts with the analyst and continues with review by a second level reviewer who verifies that data reduction has been correctly performed and that the reported analytical results correspond to the data acquired and processed. The procedure is outlined in Figure 8.6-1.

8.6.1 Data Reduction

The analyst's responsibilities for data reduction include the following:

- Proper identification of analysis output (charts, chromatograms, mass spectra, etc.)
- Calculation of instrument linearity
- Calculation or verification of QC sample/standard results
- Calculation or verification of sample results
- Use of proper data recording procedures (as described in operation-specific SOPs)
- Documentation of problems encountered
- Reporting of any nonconformances
- Continuation of internal chain-of-custody, if applicable

In general, data will be processed by an analyst in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets
- Input of raw data for computer processing
- Direct acquisition and processing of raw data by a computer

If data are manually processed by an analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors (RFs), dilution factors, and calibration constants. If calculations are not

performed directly on the data sheet, they may be attached to the data sheets.

For data that are input by an analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the project number and other information as needed. The samples analyzed must be clearly defined.

If data are directly acquired from instrumentation and processed, the analyst must verify that the following are correct:

- Project and sample numbers
- Calibration constants and RFs
- Units
- Numerical values used for detection limits (if a value is reported as "less than")

Analysis-specific calculations for methods are provided in operation-specific SOPs. In cases where computers perform the calculations, software must be validated or verified (if industry-standard software is used) in accordance with operation-specific SOPs before it is used to process data. Software validation and verification is discussed in Section 6.0.

The analyst (initial reviewer) is further required to initiate a data review check list for each batch of samples. Data review check lists are described in the ITAS Division SOP No. ITAS-IT-QC-0003, "ITAS Data and Report Review Responsibilities". ITAS data review check lists are shown for the following analyses:

- Metals (Figure 8.6-2)
- General Chemistry (Figure 8.6-3)
- Air (Figure 8.6-4)
- Radiochemistry (Figure 8.6-5)
- GC/MS (Figure 8.6-6)
- GC (Figure 8.6-7)
- HPLC (Figure 8.6-8)
- Dioxins/Dibenzofurans (Figure 8.6-9)

Data review check lists define and document the reviews that are performed on analytical data. The data review check list contains the specific items to be verified for the applicable test method. The items in the check list must be addressed by the initial reviewer (analyst), who must add to the check list any comments that should be relayed to the Project Manager for inclusion in the case narrative. The signature of the reviewer on the check list will serve to document that the initial data verification has been performed.

8.6.2 Data Verification

Following the completion of the initial

review by the analyst, a systematic second-level review of the data is performed by an experienced peer, technical person, or supervisor. The second level reviewer examines the data using the appropriate check list signed by the analyst. This review includes at least 20% of all items listed and in some cases up to 100% of all items (e.g. if mistakes are found in the 20% review). Any exceptions noted by the analyst must be reviewed. Included in this review is an assessment of the acceptability of produced data with respect to:

- Adherence of procedure used to the requested analytical method or SOP
- Correctness of numerical input when computer programs are used
- Numerical correctness of calculations
- Correct interpretation of chromatograms, mass spectra, etc.
- Acceptability of QA/QC data
- Documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.)
- Documentation of dilution factors, standard concentrations, etc.

This review also serves as verification that the process that the analyst has followed is correct in regard to the following:

- The analytical procedure follows the methods and specific instructions given in the QAS, QAPjP, SOP, and/or project file
- Nonconforming events have been addressed by corrective action as defined in a nonconformance memo
- Relevant comments about sample or analysis problems are clearly stated
- Valid interpretations have been made during the examination of the data and the review comments of the initial reviewer are correct
- The package contains all of the necessary documentation for data review and report production, and results are reported in a manner consistent with the method used for preparation of data reports

The specific items covered in the second-level review may vary according to the analytical method, but this review of the data must be documented on the data review check list with the signature of the person performing the review.

A third-level review is performed by the Project Manager. This review is required before results are submitted to clients. This review serves to verify the completeness of the data report and to ensure that client project requirements are met for the analyses performed. The items to be reviewed are:

- Analysis results are present for every sample in the analytical batch or sample delivery group

- Every parameter or target compound requested is reported with either a concentration of detection limit

- The correct units and correct number of significant figures are given for each sample and each parameter reported

- If specific data reporting forms were requested, all forms are present and are completed correctly

- All nonconformances and data evaluation statements that impact the data quality are accompanied by clearly expressed comments from the laboratory

- The final report is legible, contains all the supporting documentation required by the project, and is in either the standard ITAS format or in the client-required format

A case narrative to accompany the final report will be prepared by project management. This narrative will include relevant comments from the earlier reviews as determined by the Project Manager.

8.6.3 Data Reports

The format and content of data reports are dependent upon client needs such as client or contract requirements and government agency reporting formats. There are three general categories of data reports:

- Certificate of Analysis
- Data Package
- Electronic Data Transfer (EDT)

8.6.3.1 Certificate of Analysis

ITAS laboratories report data in a standard format, unless client or contract requirements take precedence. Figure 8.6-10 shows pages one and two of the standard ITAS Certificate of Analysis.

On page one, client/project information is presented such as:

- Client name and address
- Date
- Job Number
- Purchase Order number
- Project Identification
- Date samples received
- Number and type of samples

After the client/project information, explanatory text begins with an

Introduction. The Project Manager or designee signs page one after reviewing and approving the report.

Page two continues the explanatory text section with an Analytical Results/Methodology section and a Quality Control section. Following page two is a presentation of the results of the testing program along with QC sample summaries as appropriate.

Data presentation includes:

- Sample identification (both client and laboratory ID)
- Parameter(s) analyzed
- Reported values
- Units of measurement
- Detection limit(s)
- Explanation of abbreviations used, if applicable
- Dates of extractions/digestions and analysis
- QA/QC data (if requested by the client)

8.6.3.2 Data Package

ITAS routinely prepares data packages in accordance with USEPA CLP protocol for samples analyzed according to the CLP Statement of Work. "CLP-like" data packages are also routinely provided for non-CLP analyses. Data packages shall be prepared to meet client and contractual needs. Special data package requests should be addressed with the laboratory prior to sample analysis to assure the proper protocol is followed to generate the needed data package elements.

In general, data packages consist of a case narrative followed by computer-generated data forms, supported by copies of all associated raw data. This may include but not be limited to copies of:

- Chromatograms
- Extraction notes
- Digestion logs
- Strip charts or instrument computer printouts
- Data worksheets
- Standards logs
- Chain-of-Custody records

8.6.3.3 Electronic Data Transfer (EDT)

Upon request, data may be transmitted via computer or diskette. Each operating unit will work with the client to provide a computer format that is consistent with the output of laboratory data and meets contractual needs. Procedures for EDT are documented in operation-specific SOPs.

8.6.3.4 Verbal Results

Any analytical results communicated verbally or by facsimile should be reviewed and approved prior to the communication. Thus, these results must be of the same quality as the hard copy report.

8.6.3.5 Data Reporting

Sample results are reported per analytical method SOPs or per contract specification. Normally, the laboratory determines a reporting limit at which any analyte of interest detected at or above that level is reported as a positive value and any analyte of interest not detectable or detected below that level is reported as a "less than" value. However, in some cases a situation may occur, due to a contractual requirement, QAPjP, QAPP, client request, etc., that requires the laboratory to report sample results in a specified manner. Some examples are given below:

- The laboratory may be requested to report all analytes of interest that are less than the laboratory's reporting limit but are measurable by the analysis. This data will be flagged with an appropriate qualifier.

- The laboratory may be requested to report any tentatively identified compounds less than or greater than the laboratory's reporting limit. This data will be flagged with an appropriate qualifier.

- The laboratory may be requested to report sample results using a reporting limit that is higher than their normal level. In this case, all analytes of interest not detected or detectable below that level would be reported as "less than" and only the analytes of interest found at or above that level would be reported as positive values. In this case, the laboratory will state the specified reporting level rather than their normal levels.

In these types of cases, the laboratory must include documentation in the project file that supports their reporting procedure.

It is the responsibility of the laboratory to provide for a reporting system that ensures that any problems associated with an analysis are properly documented on a nonconformance memo, communicated to the appropriate ITAS Associates, and addressed appropriately in the data report.

8.7 Data Validation

In ITAS and this OS QAMP, data validation refers to data reviews conducted in accordance with the USEPA CLP "Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses" and "Laboratory Data Validation Functional Guidelines for Inorganic Analyses". Data validation performed by ITAS will be done according to ITAS Division SOP No. ITAS-IT-DV-001, "Quality Assurance Plan - Data Validation".

This form of data validation provides an impartial confirmation of the laboratory's results. It is usually performed by a third party. Qualifiers are assigned to data, when required, per the above mentioned documents. The ITAS FAS units routinely perform data validation services.

8.8 Preventive Maintenance

Instruments, equipment, and parts are subject to wear, deterioration, or change in operational characteristics. Within ITAS, preventive maintenance is an organized program of actions taken to maintain proper instrument and equipment performance. The primary purpose of this program is to prevent instrument and equipment failure and minimize down time. A properly implemented preventive maintenance program increases the reliability of a measurement system.

Each instrument or piece of equipment shall be uniquely identified. Each operating unit shall maintain the following:

- Instrument/equipment inventory list
- Instrument/equipment major spare parts list or inventory
- External service contract documents (if applicable)
- Instrument-specific preventive maintenance logbook or file for each functional unit

The record of maintenance shall include at a minimum:

- Actions taken, including parts replaced
- Analyst initials and date maintenance was performed

ITAS documents and describes in detail instrument/equipment preventive maintenance in operation-specific SOPs. SOPs are specific to the type of instrument or equipment being used for sample analysis.

8.8.1 Responsibilities

Within each laboratory, the Operations Manager is responsible for overseeing the preventive maintenance program. Group Leaders and Analysts actually implement and document the program. The QA/QCC shall review implementation to verify compliance.

8.8.2 Frequency of Maintenance

The frequency of maintenance must consider manufacturer's recommendations and previous experience with the instrument or equipment. Schedules of preventive maintenance along with the required frequency are shown in Appendix Section 7.

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9.0 QUALITY ASSESSMENT AND RESPONSE

Each ITAS operating unit shall establish, implement, and document procedures to detect, prevent, and correct quality problems and to ensure quality improvement. Items and processes that do not meet established requirements must be investigated to determine their cause. Improvements must be implemented in the operations which will prevent a recurrence of these quality problems and provide overall quality performance. All phases of laboratory work should be designed with the objective of preventing problems and improving quality on a continuous basis.

9.1 Internal QC

The quality of all data produced at ITAS operations is demonstrated by the analysis of required QC samples in addition to the specified method performance criteria such as calibrations. Operation-specific SOPs include information on all requirements for the type of QC samples, their target frequencies, and target acceptance criteria for each analytical methodology to be used. Additionally, these SOPs describe the appropriate actions to be taken when a QC sample result does not meet target acceptance criteria. This information is listed in Table 8.5.1 for inorganic QC samples and Table 8.5-2 for organic QC samples.

9.2 Specific Routine Procedures Used to Assess Data Precision, Accuracy, and Completeness

Section 8.5 of this document describes the QC samples that are employed at ITAS as a routine part of sample analysis. The results of these QC samples will be used to validate the precision and accuracy of the laboratory measurements. QC samples are split into two categories: Laboratory Quality Control Measurements and Matrix Quality Control Measurements. These are described in the following sections:

9.2.1 Laboratory QC Measurements

Laboratory QC samples (discussed in section 8.5.3) demonstrate that the functions which are under laboratory control are within acceptable limits. Table 9.2-1 lists laboratory QC samples and their purpose.

9.2.2 Matrix QC Measurements

Matrix QC samples provide information regarding any influence the sample matrix may exert on the precision and accuracy of the analytical results. As this influence is beyond the control of the laboratory, matrix QC samples outside the acceptance criteria are not always cause for re-analysis of the sample. Matrix QC samples include

laboratory MS, MSD, and duplicate samples. Matrix QC samples and their purpose are listed in Table 9.2-2.

9.2.3 Precision and Accuracy Limits

Precision and accuracy limits are defined in the applicable methods and will be used to determine the acceptability of the QC sample results. In the case where a method does not specify precision and accuracy limits, internal limits will be determined by the ITAS operating unit and documented in operation-specific SOPs.

Table 9.2-3 shows the precision and accuracy measurements employed by ITAS. MS, MSD, and duplicate sample results are evaluated using method or laboratory limits. Control charts or tables are maintained by the analyst for LCSs on a "real time" basis and are used to assess the level of laboratory control. It is the responsibility of each laboratory analyst to update any other client-required control charts or control tables as a part of routine data reduction.

9.3 Nonconformance, Corrective Action, and Deficiency

9.3.1 Nonconformance

A nonconformance is a deviation or event beyond the limits and criteria established for standard operations, which may lead to a degradation of quality to an unacceptable or indeterminate level. Nonconformances over which the operating unit has control must have the root cause determined so that the possibility of the nonconformance recurring is minimized or eliminated.

Nonconformances may include (but are not limited to) the following:

- Sample holding time exceeded
- Incorrect sample preparation or analysis techniques used
- Invalid instrument calibration used
- QC sample data (blank, spike, duplicate, surrogates, LCS, etc.) are outside acceptance criteria.
- Incorrect data reported to the client
- Sample lost during extraction/analysis; no re-prep or re-analysis possible
- Any other situation that might adversely affect the final data quality

As soon as deviation from accepted laboratory practice is discovered, it is required to be documented. There are two types of documentation within the ITAS system for nonconformances. These are the ITAS Nonconformance Memo (NCM), Figure 9.3-1 (pages 1 and 2) and the ITAS Condition Upon Receipt Variance Report (CUR), Figure 8.3-1.

9.3.1.1 Nonconformance Memo (NCM)

Information to be documented on the NCM include (but are not limited to) the following:

- Laboratory area affected
- Nonconformance category
- Client notification
- Corrective action (root cause and actions to prevent recurrence should be included, as appropriate)
- QC review (nonconformance or deficiency must be checked off)
- Verification of corrective action if nonconformance (verification is not required for deficiencies)
- Signature by QA/QCC or designee verifying NCM closure
- Routing destination (Quality/Operations files or Project file)

Upon completion, original NCMs are stored in the appropriate project file if project-specific or are stored in the facility Quality/Operations files if non-project specific.

Nonconformances identified through surveillance, or through an internal or external audit require documentation and tracking to verify that the root cause has been determined and that corrective actions have been taken to prevent recurrence. The NCM may be used for this purpose.

9.3.1.2 Condition Upon Receipt Variance Report (CUR)

A CUR is generated by sample control during the log-in process to document nonconformances identified upon receipt of samples in the laboratory. These nonconformances are outside of laboratory control and do not require corrective actions to be taken within the laboratory. The corrective action in this case is client notification. Actions to prevent recurrence should take the form of client education when possible. Nonconformances documented on a CUR may include (but are not limited to) the following:

- Not enough sample received for proper analysis
- Sample received without proper preservative
- Sample received in improper container
- Sample received broken or leaking
- Sample received outside temperature specifications
- Sample received without proper paperwork
- Sample received without or with broken custody tape

- Chain-of-Custody broken (not relinquished by the client)
- Sample information on the container does not match the accompanying paperwork
- All shipping containers (coolers) on waybill not received in the sample shipment

Once the nonconformance is identified and the CUR initiated, the laboratory Project Manager is notified. The Project Manager then notifies the client and requests instructions on how to proceed with the samples. The client contact and the client response to the nonconformance must be documented on the CUR and communicated to sample control so that log-in may proceed. These nonconformances must be resolved prior to sample prep and analysis.

9.3.2 Corrective Action

All nonconformances shall have a corrective action. The process by which corrective action is performed requires a determination of root cause, immediate action, and actions taken to prevent recurrence. The latter two may be the same action. Corrective actions include (but are not limited to) the following:

- Recalibration of instruments, using freshly prepared calibration standards
- Reanalysis of samples
- Replacement of lots of solvent or other reagents that yield unacceptable blank values
- Additional training of laboratory Associates in correct implementation of sample preparation and analytical techniques
- Reassignment of Associates
- Communication with the client to determine appropriate action (e.g. resampling, processing the sample "as is", terminating analysis, etc.)

9.3.3 Deficiency

A deficiency is a deviation from documented procedures, practices, standards, or a defect in an item that is determined *not* to render the quality of an item or service unacceptable or indeterminate. The QA/QCC shall determine whether the deviation is a nonconformance or a deficiency.

9.3.4 Responsibilities

All laboratory Associates are responsible for identifying and reporting any deviation from accepted laboratory practice that might affect the quality of the data. Once a possible nonconformance is identified, a NCM or a CUR is generated and routed to that Associate's supervisor for further review and documentation.

The QA/QCC is responsible for verifying

corrective actions and tracking NCM's until closure. The corrective action must be performed in a timely manner.

The QA/QCC (or designee) is responsible for logging all nonconformances into a master nonconformance log (example shown in Figure 9.3-2).

9.4 QA/QC Audits

ITAS operating units are subject to numerous assessments in the form of audits, both internal (self assessment) and external (independent assessment). Laboratory audits within ITAS can be broken down into four major categories:

- Performance Audits
- Surveillances
- Quality Systems Audits
- Project Audits

Audits of laboratories are performed to determine the degree of adherence to policies, procedures, and standards which include:

- IT and ITAS Quality Assurance Policy
- IT and ITAS Procedures
- Contractual Requirements
- Regulatory Obligations

Audits serve as a useful management tool to evaluate the appropriateness of QA policies. They identify areas for improvement with regard to compliance with policies, procedures, and standards, providing means for correction prior to system failure requiring shut down. In addition, they serve to strengthen the documentation trail assuring known data quality.

9.4.1 Performance Audits

Performance audits are conducted on an ongoing basis within the laboratory by the QA/QCC. These audits are reported to the Operating Unit Director and the Division Director, QA/QC. Performance audits vary with the needs of the operating unit. Performance audits include internal and external performance evaluation (PE) samples. These are discussed in the following sections.

9.4.1.1 Internal Performance Evaluation

The QA/QCC has the responsibility of monitoring the performance of the laboratory by inserting blind QC samples ("true" value(s) unknown to analyst) into the sample stream periodically and analyzing the results. The blind QC samples will be scheduled throughout the year to cover all routine analyses on an annual basis. They may also be performed

along with a project (client requested) or any time the QA/QCC or Operating Unit Director requests the performance audit study. Performance audit samples demonstrate data quality by statistical analysis. The results of these samples may also be used to document the training level of the analyst(s) performing the work. These results are linked to the analyst(s) in the Associate training files.

9.4.1.2 External Performance Evaluation

Each laboratory participates in external PE programs such as the USEPA Water Supply (WS), Water Pollution (WP), and/or Contract Laboratory Program (CLP) quarterly proficiency program for chemical parameters and/or the USEPA Intercomparison Program through the Environmental Monitoring Support Laboratory in Las Vegas (EMSL-LV) for radiochemical parameters. In addition, many state agencies and private contractors provide PE samples to challenge the laboratories and evaluate the effectiveness of the laboratory program. All external PE sample studies and results shall be maintained as quality records in the laboratory Quality/Operations files.

Since participation in these programs and others varies with the type of work performed within each ITAS laboratory, the specifics of participation are described in Appendix Section 6.

9.4.1.3 Double-Blind Performance Evaluation

ITAS employs a "double blind" PE sample program involving semiannual studies. This program applies to all ITAS laboratories. Double-blind PE samples are blind PE samples submitted to the laboratory under the pretense that they are normal client samples. The results of the study are reported to the Vice President, IT Analytical Services, the Division Technical Director, and the Division Director, QA/QC. Recommendations for quality improvement are submitted to the Laboratory Directors, and corrective actions are implemented as necessary.

9.4.2 Surveillances

Surveillances are detailed inspections of specific areas of a laboratory and its QA Program. Surveillances do not require the extensive planning and preparation required for audits and are conducted on a much more informal basis. The QA/QCC shall observe the activity of interest while it is in process and/or review objective evidence. A checklist for the applicable documents and criteria may be used for this review.

corrective actions and tracking NCM's until closure. The corrective action must be performed in a timely manner.

The QA/QCC (or designee) is responsible for logging all nonconformances into a master nonconformance log (example shown in Figure 9.3-2).

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are performed on a routine basis and are described in operation-specific SOPs. In addition, they may also be performed along with a project (client requested) or any time the QA/QCC or Operating Unit Director requests the performance audit study. Performance audit samples demonstrate data quality by statistical analysis. The results of these samples may also be used to document the training level of the analyst(s) performing the work. These results are linked to the analyst(s) in the Associate training files.

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Surveillances are detailed inspections of specific areas of a laboratory and its QA Program. Surveillances do not require the extensive planning and preparation required for audits and are conducted on a much more informal basis. The QA/QCC shall observe the activity of interest while it is in process and/or review objective evidence. A checklist for the applicable documents and criteria may be used for this review.

A surveillance is performed each month by the QA/QCC (unless a systems audit or follow-up audit is performed). The scope of the surveillance is determined by the QA/QCC. This allows for a concentrated focus on areas of the laboratory that may be suspect or require additional monitoring to verify compliance with policies, procedures, and standards. The QA/QCC may use the nonconformance/corrective action system to determine trends in a laboratory area that require further investigation. The purpose of a surveillance is to find and correct problems before they become out-of-control situations.

Once the surveillance is complete, the QA/QCC will assign a score and will issue a report to the responsible manager. A copy of the report is sent directly to the Laboratory Director and to the Division Director, QA/QC in the laboratory monthly report to management. The report details the results of the surveillance, and requests a corrective action plan complete with target dates and Associate assignments. The QA/QCC must work with the surveyed group to recommend corrective action and then follow-up after the proposed target date to verify that corrective action was indeed performed. The QA/QCC shall document by memorandum that corrective action was taken. Surveillances are fully described in operation-specific SOPs.

9.4.3 Quality Systems Audits

Four times per year, each laboratory undergoes an internal audit to identify the level of compliance with established, documented, quality assurance systems. Two of these audits are designed and conducted by the QA/QCC. The remaining two are conducted by the Division Director, QA/QC or designee. The Division audits usually consist of a 2-4 day comprehensive review of all quality systems in the laboratory. Six months later, a second or follow-up audit is conducted to assess compliance with the corrective action plan established by the audited laboratory after completion of the first audit. The follow-up can be performed in 1-2 days. The Lead Auditor reserves the right to lengthen the audit or require a complete re-audit in 3 to 6 months depending upon the extent of the problems discovered. Findings which have not been satisfactorily resolved between the two audits shall be specifically reported to the Division Operations Director and Vice President, IT Analytical Services, for resolution.

Systems audits not conducted by the QA/QCC are lead by an ITAS certified Lead Auditor (see System Procedure 8907-QAC-04, "Standard Operating Procedure for Auditors Certification at ITAS Laboratories") under the direction of the Division Director, QA/QC. The Division

Director, QA/QC (or designee) will prepare a schedule of audits to be conducted during the fiscal year and will select appropriate audit teams depending upon the nature and depth of the audit. The source documents for systems audits are the ITAS QAMP, the ITAS Operation-Specific QAMP, and SOPs. The scope of the audit takes into account the expectations of external auditors, contracts, and regulatory requirements. A systems audit check list is prepared for the year and used in all locations audited to provide consistency and objectivity to the audit team.

At the beginning of the audit, the audit team will meet with the Operating Unit Director and the QA/QCC to discuss the goals of the audit. At the close of the audit, the audit team will debrief the Operating Unit Director, QA/QCC, the laboratory Technical Director, Project Managers, and Group Leaders, and will discuss and present the audit findings and observations. Additional laboratory staff may be invited to the debriefing, as deemed necessary by the Lead Auditor. The Lead Auditor can close an audit finding or observation during the debriefing if the laboratory staff can satisfactorily demonstrate that the finding/observation is inappropriate or has been corrected prior to the debriefing. Also during this meeting, recommendations for corrective actions will be discussed. If corrective actions are requested to be taken immediately after audit closure, the actions must be taken.

An audit report will be prepared by the Lead Auditor and will include the following:

- Cover memo summarizing the audit process, any findings, and announcement of the preliminary audit ranking
- Audit Check List
- Finding Report(s)
- Observation Report(s)
- Corrective Action Plan (to be completed by the laboratory)

The audit report shall be completed as soon as possible after completion of the audit, but shall take no longer than 45 days. The original audit report will be addressed to the Operating Unit Director, who is responsible for responding within the designated time frames established by the Lead Auditor. A completed copy of the systems audit report will be sent to the QA/QCC and the Division Director, QA/QC.

Upon receipt of the audit response, the Lead Auditor will evaluate the proposed corrective action plan and will reply stating acceptance or rejection of the plan or its

elements. Approximately six months later, the Lead Auditor or designee will perform a second audit of the operation and verify completion of the initial audit corrective action plan. The Lead Auditor will then issue a final audit report detailing the results of implementation and will issue a final audit ranking for the year. A positive change in ranking is indicative of improvement in implementation of the QA Program.

9.4.4 Project Audit

Project or data quality audits are designed to address the DQOs (precision, accuracy, representativeness, and completeness) of all data associated with a particular project. These audits also review a project for compliance with contractual requirements set forth in a QAPjP or formal contract. Project audits may be conducted by IT, ITAS, or project QA staff. A project-specific check list is prepared using the QAPjP and/or contract as the source document(s). The audit report is addressed to the Operating Unit Director who is required to respond within the designated time frame stipulated by the Lead Auditor. As with all other audits, a follow-up audit for verification of compliance with the corrective action plan is to be performed.

9.4.5 Findings, Observations, Comments, and Recommendations

Findings represent areas in which the operation or operating unit section as a system is not in compliance with the requirements of the ITAS QA Program. Findings are situations that could directly affect the quality of resulting work. Findings require that a corrective action plan be developed by the Operating Unit Director, who will identifying the root cause of the problem and will schedule action to prevent recurrence.

Observations represent isolated instances of noncompliance or questionable practices. They present situations that could become findings if left unresolved. As with findings, a corrective action plan is required.

Comments or recommendations shall be written by the auditor in an attempt to share information and provide constructive criticism in order to improve performance or documentation in an area. Comments might also indicate areas that may become noncompliant. If attention is not paid to the comment, it is likely to become an observation. Comments and recommendations do not require any formal response by the audited organization, but it is strongly recommended that they be reviewed for appropriate action. Included in

the comments and observations are those which describe exemplary practices. Audit reports should not focus only on negative aspects of the program and may include a section on exemplary practice.

9.4.6 Audit Ranking

Internal ITAS QA Systems Audits require a preliminary and final ranking to be assessed. Audit check list scores and assessments of open observations and findings (not corrected since the last internal or client audit) are considered in the audit ranking process. Laboratories are ranked as either excellent, acceptable, marginal, or unacceptable. These ranks are described as follows:

- Excellent - Meets or exceeds established requirements for all areas audited.
- Acceptable - Audited work meets all requirements of the ITAS QA Program with only a few minor deviations from established requirements.
- Marginal - Audited work represents a basic QC practice with actions required by the laboratory to improve operations immediately.
- Unacceptable - Audited work indicates that quality practice is not implemented on a regular basis and one or more areas will be shut down for correction.

The Division Director, QA/QC, will issue a memorandum to all QA/QCCs and Operating Unit Directors annually describing the process of determining the audit rank.

While audit ranking allows for comparison of laboratories across the network, caution is advised in using the ranking alone without a detailed review of the situations or conditions observed that caused the Lead Auditor to arrive at that rank.

9.4.7 Client Satisfaction Survey

Each ITAS operating unit has the responsibility to understand client needs, and whether ITAS services are meeting those needs and expectations. At least three client satisfaction surveys should be performed monthly, within each operating unit, to randomly selected clients by the QA/QCC or designee. The IT Corporation Client Satisfaction Survey form (Figure 9.4-1) should be utilized with distribution to the ITAS Project Manager, Operating Unit Director, Division Operations Director, Vice President, IT Analytical Services, Vice President, Quality and Health Services, and Division Director, QA/QC. Any corrective or follow-up action must be documented on the form and implemented by the Project Manager or Operating Unit Director.

9.5 Quality Reports to Management

The QA/QCC and the Division Director, QA/QC shall prepare reports to

elements. Approximately six months later, the Lead Auditor or designee will perform a second audit of the operation and verify completion of the initial audit corrective action plan. The Lead Auditor will then issue a final audit report detailing the results of implementation and will issue a final audit ranking for the year. A positive change in ranking is indicative of improvement in implementation of the QA Program. Quality systems audits are described in a operation-specific SOPs.

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9.5 Quality Reports to Management

The QA/QCC and the Division Director,

management on a monthly basis indicating the effectiveness of the QA Program. The QA/QCC shall send a quality report to the Division Director, QA/QCC. An example outline for the QA/QCC's report is shown in Figure 9.5-1. The Division Director, QA/QC, shall send a summary of the QA/QCC's reports to the Vice President, IT Analytical Services.

9.6 Management Review of the Quality Assurance Program

Management at all levels shall assess the QA Program and its performance. Management assessment shall identify barriers that hinder the organization from achieving its objectives in accordance with quality, safety, and environmental requirements. Results of management assessments shall be documented and corrective action taken. The effectiveness of the implementation of corrective actions shall be included in the next management assessment.

An example of the management assessment approach will be to conduct double blind studies on the appropriate laboratories (see Section 9.4.1.3). In such studies, a client contacts the laboratory and submits a sample of known parameters and values for analysis that is totally blind to the laboratory management and the analysts. These studies allow assessment of the total process from initial client contact through final reporting.

Review of the adequacy of the ITAS QA Program is ongoing. At any time, the Division Operations Director, an Operating Unit Director, or the Division Technical Director may present, in writing, recommended changes to the Division Director, QA/QC. During the QA systems audits, the QA Program is discussed with the management of the facility audited. This feedback is valuable and necessary to the progress of the QA Program in meeting the constantly-changing needs of the environmental industry.

In addition to these ongoing reviews, the Vice President, IT Analytical Services shall conduct an annual review of the QA Program considering:

- Results of the QA systems audits. Are undesirable trends occurring?
- Status of QA documents. Are the current documents adequate? Are new documents needed?
- Is the auditing program fulfilling its purpose?

The Vice President will consult with the Division Operations Director, Operating Unit Directors, and the Vice President, Quality and Health Services, as necessary, during the review. To document the review, the Vice President, IT Analytical Services,

will issue a memo to the Division Director, QA/QC, stating the extent of the review and will present recommendations.

FIGURE 1.2-1
INTERNATIONAL TECHNOLOGY CORPORATION
ANALYTICAL SERVICES
DIVISION OPERATIONS

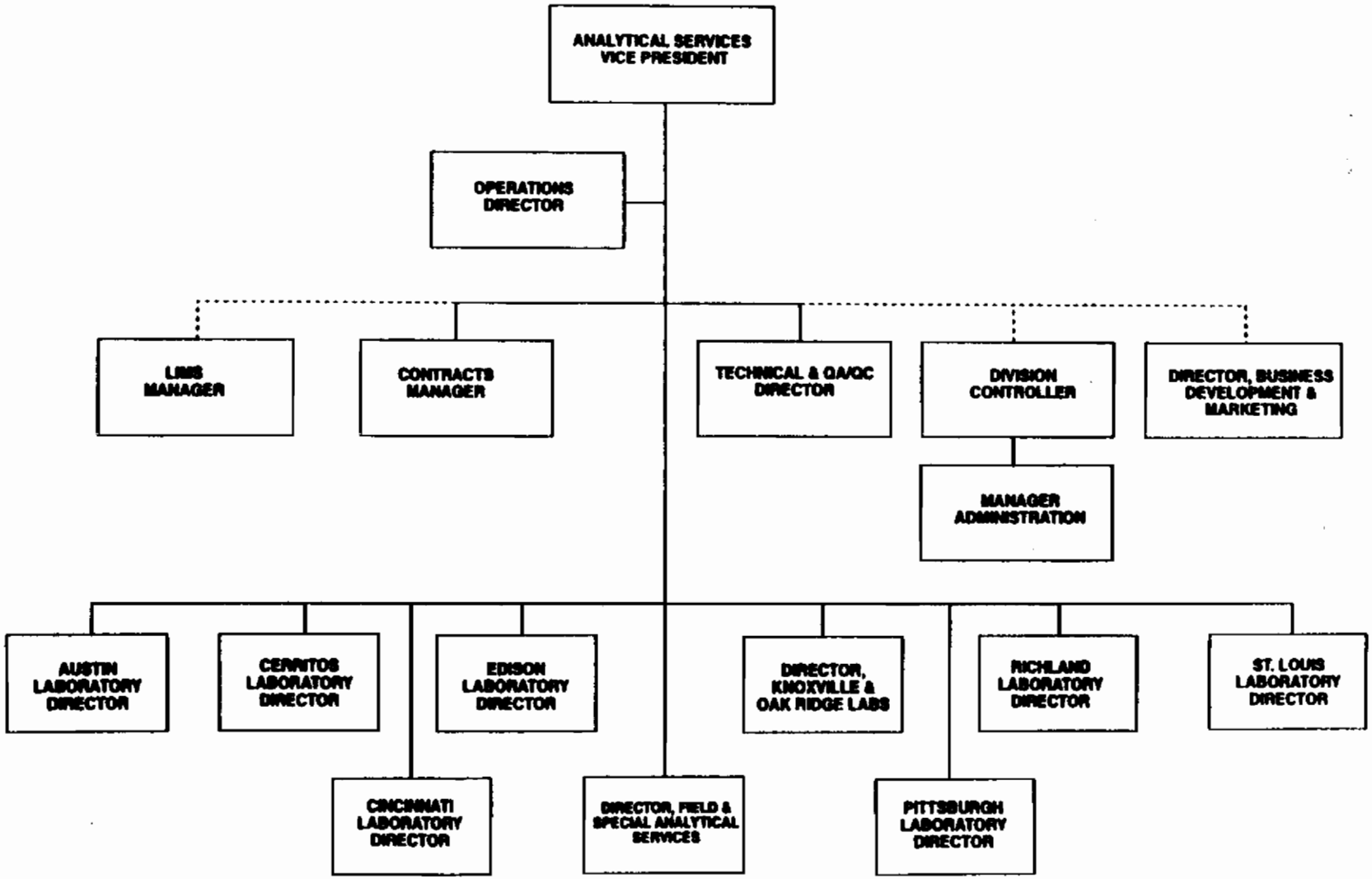

FIGURE 3.1-1
IT Analytical Services
Example Associate Qualification Form

Name: _____ **Hire Date:** _____

Title: _____ **Supervisor:** _____

PROCEDURE NAME/NUMBER	QUALIFY DATE	APPROVAL SIGNATURE

TRAINING SESSION	DATE	APPROVAL SIGNATURE

FIGURE 3.2-1
Example Individual Training Record

ne: _____ Hire Date: _____

e: _____ Supervisor: _____

I. ACADEMIC TRAINING/DEGREES

Name of Institution (Including Current Enrollment)	City, State	Curriculum Major	Attendance	Degree(s) Received/Expected	Degree Date

II. SPECIAL SCHOOLING/TRAINING

Date(s)	Course Title	Sponsored By (IT or External)/Location

III. SPECIALIZED EXPERIENCE/ON-THE-JOB TRAINING

Date(s)	Specialized Experience	Reference (Paper or Project)

ociate Signature/Date: _____

Concurrence/Date: _____

NREC.FRM\pbc\07-18-93

FIGURE 3.2-2

Example ITAS New Employee Quality Assurance Orientation Form

Name: _____ Date of Hire: _____

Job Title: _____ Report to Work Date: _____

QUALITY ASSURANCE PROGRAM SECTION	REVIEW (X)
Statement of Management Position	
ITAS Quality Assurance Management Plan (QAMP)	
Operation-Specific Quality Assurance Management Plan (OS-QAMP)	
Nonconformance and Corrective Action	
Data Entries and Corrections	
NQA-1	
NRC Regulatory Guide	
Quality-related responsibilities for Job Title (Section and/or Topic):	
_____	

I attended the session covering QA sections and/or topics as described above and understood the material presented during the session.

_____ _____

Associate's Signature Date

_____ _____

QA/QC Coordinator's Signature Date

QA Exam Given? _____ Orientation was adequate _____ Further training is needed _____

Follow-up sessions covered:

_____

_____

_____ _____

Associate's Signature/Date QA/QC Coordinator's Signature/Date

THIS DOCUMENT MUST BE RETAINED IN THE ASSOCIATE'S TRAINING FILE

FIGURE 3.2-3

Example ITAS Lead Auditor Certification Form

NAME	LOCATION	DATE

QUALIFICATION POINT REQUIREMENTS		CREDITS
EDUCATION - University/Degree/Date	4 Credits Max.	
1. Undergraduate Level		
2. Graduate Level		
EXPERIENCE - Company/Dates	9 Credits Max.	
1. Technical (0-5 credits)		
2. Nuclear Industry (0-1 credits) OR		
3. Analytical (Environmental) Industry (0-1 credit) OR		
4. Quality Assurance (0-3 credits) OR		
5. Auditing (0-4 credits)		
PROFESSIONAL ACCOMPLISHMENT - Certificate/Date	2 Credits Max.	
1. Society		
2. Other		
MANAGEMENT - Justification/Evaluator/Date	2 Credits Max.	
Explain:		
Evaluated By: (Name & Title) Date:		
	Total Credits	0

AUDIT TRAINING/PARTICIPATION

AUDIT COMMUNICATION SKILLS

Evaluated By: (Name & Title) Date:

AUDIT TRAINING COURSES

Course Topic or Title
1
2

AUDIT PARTICIPATION

	Location	Type of Audit	Date(s)
1			
2			
3			
4			
5			
6			
7			

EXAMINATION Passed Date:

AUDITOR QUALIFIED CERTIFIED BY: **DATE CERTIFIED:**

(Signature & Date)

ANNUAL EVALUATION				
(Signature & Date)				

FIGURE 5.0-1
Example Procedural Change Form

ITAS _____ LABORATORY

PROCEDURE/DOCUMENT CHANGE

PROCEDURE/DOCUMENT TITLE AND NUMBER:

PROCEDURE/DOCUMENT SECTION(S) AFFECTED BY CHANGE:

REASON FOR ADDITION OR CHANGE:

CHANGE EFFECTIVE FROM: _____ (DATE) TO: _____ (DATE)

SAMPLES OR PROJECTS AFFECTED:

CHANGE OR ADDITION (SPECIFY SECTION; USE ADDITIONAL SHEETS IF NECESSARY):

SUBMITTED BY: DATE:

QA/QC COORDINATOR

LABORATORY DIRECTOR

FIGURE 5.4-1
ITAS Standard Administrative SOP Format

1. Purpose, Application and Responsibility
2. References
3. Associated SOPs
4. Definitions
5. Procedure
   - 5.1 Summary
   - 5.2 Safety
   - 5.3 Required Equipment
   - 5.4 Administrative Procedure
   - 5.5 Calculations
   - 5.6 Quality Control
6. Nonconformance and Corrective Action
7. Records Management and Documentation

Note: The inclusion of subsections 5.3 through 5.6 is left up to the discretion of the SOP's author, as appropriate.

FIGURE 5.4-2
ITAS Standard Technical SOP Format

1. Purpose, Application and Responsibility
   - 1.1 Purpose
   - 1.2 Application
   - 1.3 Responsibilities
2. References
3. Associated SOPs
4. Definitions
5. Procedure
   - 5.1 Summary
   - 5.2 Safety
   - 5.3 Interferences
   - 5.4 Preservation and Holding Time
   - 5.5 Required Equipment
   - 5.6 Reagents/Standards
   - 5.7 Calibration
   - 5.8 Analysis/Operation
   - 5.9 Calculations
   - 5.10 Quality Control
6. Nonconformance and Corrective Action
7. Records Management and Documentation

FIGURE 8.0-1
Data Collection Process Flow Chart

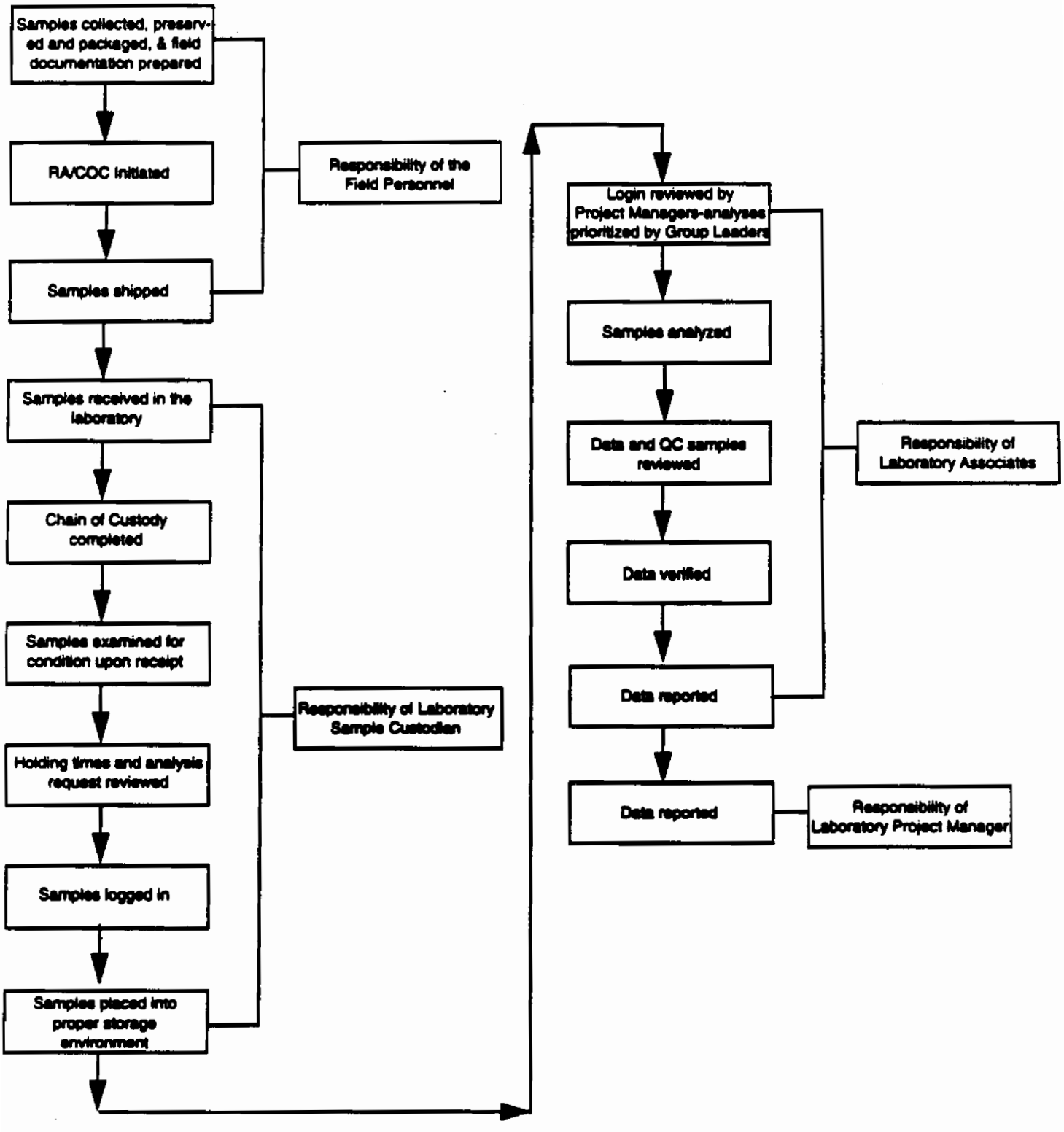

FIGURE 8.1-1

ITAS Analysis Request/Chain-of-Custody Form

INTERNATIONAL TECHNOLOGY CORPORATION

ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD*

Reference Document No. 323490
Page 1 of ____

White To accompany samples Yellow Field copy

ject Name/No. [1] _____ Samples Shipment Date [7] _____ Bill to: [5] _____

Team Members [2] _____ Lab Destination [8] _____

rofit Center No. [3] _____ Lab Contact [9] _____

roject Manager [4] _____ Project Contact/Phone [12] _____ Report to: [10] _____

hase Order No. [6] _____ Carrier/Waybill No. [13] _____

ed Report Date [11] _____

ONE CONTAINER PER LINE

mple [14] mber	Sample [15] Description/Type	Date/Time [16] Collected	Container [17] Type	Sample [18] Volume	Pre- [19] servative	Requested Testing [20] Program	Condition on [21] Receipt	Disposal [22] Record No.

al Instructions: [23]

ple Hazard Identification: [24]
zard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown ☐

Sample Disposal: [25]
Return to Client ☐ Disposal by Lab ☐ Archive (mos.)

round Time Required: [26]
d ☐ Rush ☐

QC Level: [27]
I. ☐ II. ☐ III. ☐ Project Specific (specify):

nquished by [28] (Signature/Affiliation) Date: Time:

1. Received by [28] (Signature/Affiliation) Date: Time:

nquished by (Signature/Affiliation) Date: Time:

2. Received by (Signature/Affiliation) Date: Time:

nquished by (Signature/Affiliation) Date: Time:

3. Received by (Signature/Affiliation) Date: Time:

nents: [29]

MCA 3/15/91

*See back of form for special instructions

FIGURE 8.3-1 Work Order No.:_____

Condition Upon Receipt Variance Report

ITAS - _____ Laboratory

Client:_____ Date:_____

Project No:_____ Initiated by:_____

Analysis Requested:_____ RFA/COC Numbers:_____

Client Sample Numbers Affected:_____

Condition/Variance (Check all that apply):

1. ☐ Not enough sample received for proper analysis.
   Received approximately:_____
2. ☐ Sample received broken/leaking.
3. ☐ Sample received without proper preservative.
   - ☐ Cooler temperature not within 4°C ± 2°C
     Record temperature:_____
   - ☐ pH_____
   - ☐ other:_____
4. ☐ Sample received in improper container.
5. ☐ Sample received without proper paperwork. Explain:
   _____
6. ☐ Paperwork received without sample.
7. ☐ No sample ID on sample container.
8. ☐ Custody tape disturbed/broken/missing.
9. ☐ Sample splits performed by lab.
10. ☐ Volatile sample received with approximately _____ mm headspace.
11. ☐ Sample ID on container does not match sample ID on paperwork. Explain: _____
    _____
    _____
12. ☐ All coolers on airbill not received with shipment.
13. ☐ Other (explain below):
    _____
    _____
    _____

Notes:

_____

_____

Corrective Action:

☐ Client's Name: _____ Informed verbally on: _____ By: _____

☐ Client's Name: _____ Informed in writing on: _____ By: _____

☐ Sample(s) processed "as is". Comments: _____

☐ Sample(s) on hold until: _____ If released, notify: _____

Sample Control Supervisor Review:_____ Date: _____

FIGURE 8.6-1
Data Review Process

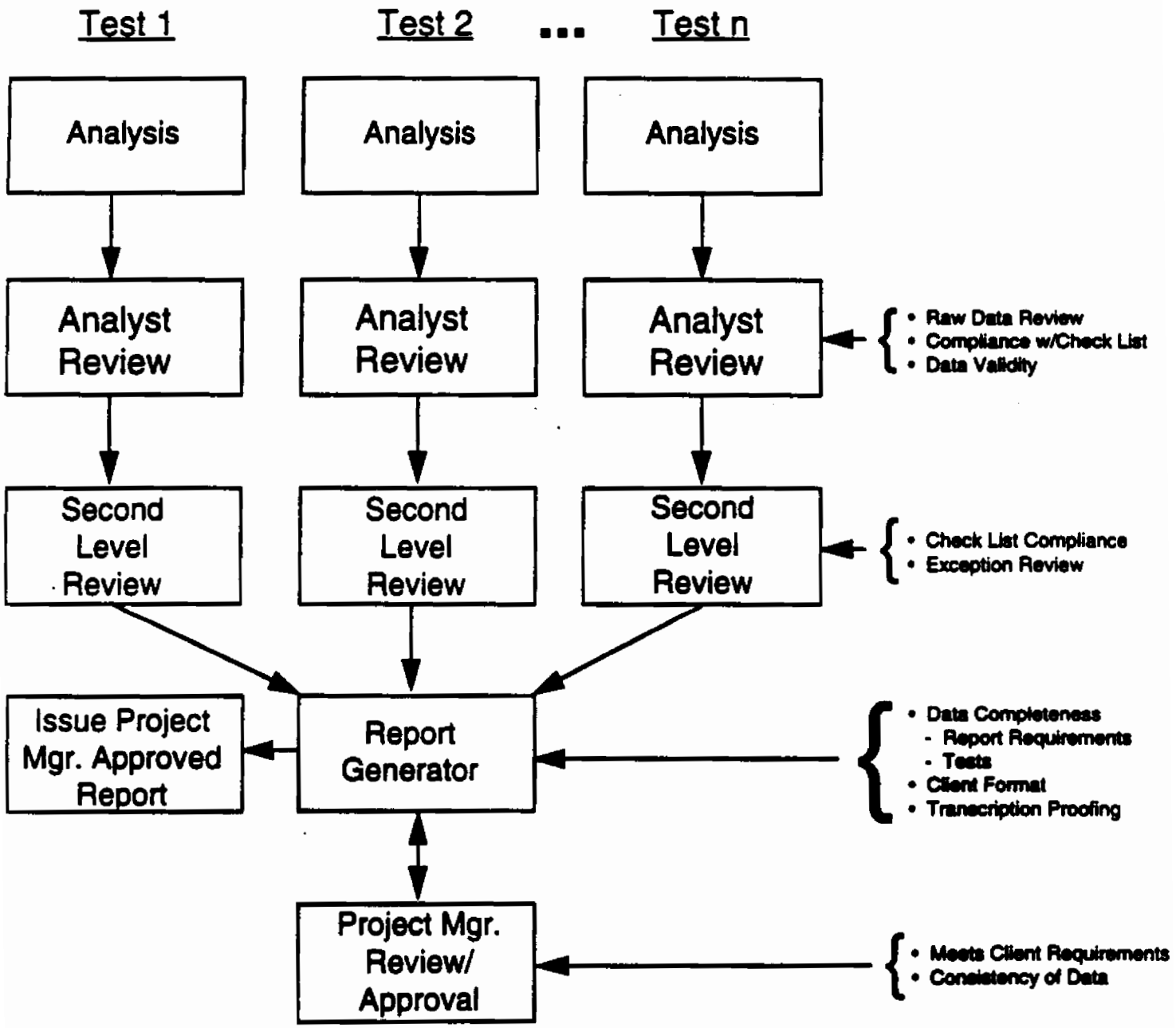

FIGURE 8.6-2
ITAS Data Review Check List
METALS

Work Order Number(s):
Lab Sample Numbers or SDG:
Method/Test/Parameter:

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Level Review (✓)
A. Initial Calibration 1. Performed at required frequency with required number of levels?				
2. Correlation coefficient within QC limits?				
3. Initial calibration verification (ICV) analyzed immediately after calibration and results within QC limits?				
4. Initial calibration blank (ICB) analyzed immediately after ICV and concentrations of all parameters ≤ reporting limit?				
B. Continuing Calibration 1. CCV analyzed at required frequency and all parameters within QC limits?				
2. CCB analyzed at required frequency and all results ≤ reporting limit?				
C. Sample Analysis 1. Were any samples with concentrations > the linear range for any parameter diluted and reanalyzed?				
2. Were all sample holding times met?				
D. QC Samples 1. MS or MS/MSD percent recovery within QC limits?				
2. Analytical spikes within QC limits?				
3. LCS recovery within QC limits?				
4. ICP only: One serial dilution performed per SDG?				
5. ICP only: CRDL standard (CRI or CRA) analyzed at required frequency?				
6. ICP only: Interference check samples (ICSA, ICSAB) analyzed at the beginning and end of analytical run or at minimum frequencies and within QC limits?				

FIGURE 8.6-2
ITAS Data Review Check List
METALS

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Level Review (✓)
E. Other 1. Are all nonconformances included and noted?				
2. Is the correct date and time of analysis shown?				
3. Did the analyst sign and date the front page of the analytical run?				
4. Correct methodology used?				
5. Transcriptions checked?				
6. Calculations checked at minimum frequency?				
7. Units checked?				

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

FIGURE 8.6-3
ITAS Data Review Check List
GENERAL CHEMISTRY

Work Order Number(s):
Lab Sample Numbers or SDG:
Method/Test/Parameter:

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Level Review (✓)
A. Initial Calibration 1. Does the standard curve consist of a Blank (when required) and the required minimum number of calibration standards?				
2. Is the initial calibration correlation coefficient ≥ 0.995?				
B. Continuing Calibration 1. Is the Continuing Calibration Verification (CCV) percent recovery within QC limits?				
C. Sample Analysis 1. Were all sample holding times met?				
D. QC Samples 1. Is the Method Blank concentration less than the reporting limit?				
2. Is the Laboratory Control Sample (LCS) AND/OR the Matrix Spike (MS) % recovery within QC limits?				
3. When MS/MSD analyzed, is RPD within QC limits?				
4. When duplicate sample analysis performed, is RPD within QC limits (± 20 %)?				
E. Other 1. Are all nonconformances included and noted?				
2. Are all required forms filled out?				
3. Was correct methodology used?				
4. Transcriptions checked?				
5. Were all calculations checked at minimum frequency?				
6. Did the analyst sign and date the front page of the analytical run?				
7. Units checked?				

FIGURE 8.6-3
ITAS Data Review Check List
GENERAL CHEMISTRY

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

FIGURE 8.6-4
ITAS Data Review Check List
AIR ANALYSIS - METHOD TO-14

Work Order Number(s):
Lab Sample Numbers or SDG:
Method/Test/Parameter:

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Level Review (✓)
A. Initial Calibration: 1. Was BFB analyzed at beginning of 12-hour period?				
2. Did BFB meet QC limits (all masses met criteria)?				
3. Is the %RSD for all nonpolar analytes <25%? (A maximum of two analytes may be above this but below 40%)				
4. Is the %RSD for all polar analytes <30%? (A maximum of two analytes may be above this but below 45%)				
5. Were a minimum of five calibration points used?				
B. Continuing Calibration 1. Was BFB analyzed at beginning of 12-hour period?				
2. Did BFB meet QC limits (all masses met criteria)?				
3. Was the %D for all CCCs <25% for all nonpolar targets? (A maximum of two are allowed outside this criteria, but must be <40%)				
4. For polar analytes, do all compounds have a %D of 30% or less? (A maximum of two are allowed outside but must be < 45%)				
5. Are all calibration points on the Continuing Calibration present?				
C. Sample Analysis 1. Are internal standard areas within 50-150% of the internal standard areas of the continuing calibration standard analyzed?				
2. Are all sample surrogate values within internal QC limits?				
3. Are all analytes quantitated within the calibrated range of the instrument?				
4. Were a minimum of three RFs checked for each sample to insure the proper RFs are being used for quantitation?				

FIGURE 8.6-4
ITAS Data Review Check List
AIR ANALYSIS - METHOD TO-14

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Level Review (✓)
D. QC Samples 1. Are all Blank surrogate values within internal QC limits?				
2. Are all analytes in Blanks < the reporting limit?				
3. Are Reference Standard control values within QC limits for air media?				
4. Are all duplicate RPDs within internal QC limits?				
E. Other 1. Are all nonconformances included and noted?				
2. Are all required forms filled out?				
3. Correct methodology used?				
4. Transcriptions checked?				
5. Were all calculations checked at minimum frequency?				
6. Units checked?				

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

FIGURE 8.6-5
ITAS Data Review Check List
RADIOCHEMISTRY

Work Order Number(s):
Lab Sample Numbers or SDG:
Method/Test/Parameter:

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Level Review (✓)
A. Calibration 1. Is the calibration documentation included where applicable?				
B. Sample Analysis 1. Are the Sample Yields within acceptance criteria?				
2. Were all sample holding times met?				
C. QC Samples 1. Is the Blank Yield within acceptance criteria?				
2. Is the Minimum Detectable Activity for the Blank result ≤ the Contract Detection Limit?				
3. Is the Blank result ≤ the Contract Detection Limit?				
4. Is the Blank result greater than the Contract Detection Limit but the Sample result less than the Contract Detection Limit?				
5. Is the LCS result within acceptance criteria?				
6. Is the LCS yield within acceptance criteria?				
7. Is the LCS Minimum Detectable Activity less than or equal to the Contract Detection Limit ?				
8. MS/MSD results and yield meet acceptance criteria?				
9. Duplicate sample results and yield meet acceptance criteria?				
D. Other 1. Are all nonconformances included and noted?				
2. Are all required forms filled out?				
3. Correct methodology used?				
4. Transcriptions checked?				
5. Were all calculations checked at minimum frequency?				

FIGURE 8.6-5
ITAS Data Review Check List
RADIOCHEMISTRY

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

FIGURE 8.6-6
ITAS Data Review Check List
GC/MS

Work Order Number(s):
Lab Sample Numbers or SDG:
Method/Test/Parameter:

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Level Review (✓)
A. Tuning 1. BFB/DFTPP tuning criteria met?				
2. Mass list, RIC, and the mass spectrum included?				
3. Correct BFB/DFTPP included with analytical runs?				
B. Initial Calibration 1. RRF and %RSD within acceptance criteria?				
2. Runs checked for saturation?				
3. CLP only: Are surrogates and internal standards labeled on the chromatograms?				
C. Continuing Calibration 1. RRF and % Difference within acceptance criteria?				
D. Sample Analysis After tune, initial calibration, continuing calibration, and method blank criteria have been met: 1. Sample name and other header information correct?				
2. RRT of identified compounds within ± 0.06 RRT units of RRT of standard component?				
3. Ions present in the standard spectra with abundance of $>10\%$ of the base ion present in sample spectra?				
4. Surrogate recoveries within limits?				
5. Quantified against the appropriate standard?				
6. Run(s) within linear range?				
7. Were all sample holding times met?				
8. TCL match?				

FIGURE 8.6-6
ITAS Data Review Check List
GC/MS

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Level Review (✓)
E. QC Samples 1. All Method Blank results ND or hits below reporting limit?				
2. Method Blank analyzed at required frequencies?				
3. Are MS/MSD % recoveries and RPD within QC limits?				
4. Are LCS % recoveries within QC limits?				
5. Second source check standard successfully analyzed?				
F. Other 1. Are all nonconformances included and noted?				
2. Are all required forms filled out?				
3. Correct methodology used?				
4. Transcriptions checked?				
5. Were all calculations checked at minimum frequency?				
6. Were all manual integrations checked by a second reviewer?				
7. Units checked?				

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

FIGURE 8.6-7
ITAS Data Review Check List
GC

Work Order Number(s):
Lab Sample Numbers or SDG:
Method/Test/Parameter:

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Level Review (✓)
A. Initial Calibration 1. Was the maximum %RSD within QC limits?				
2. Were calibration factors updated or were curves drawn for 600 series methods or 8000 series methods?				
3. Are retention time windows established and updated in method?				
4. Did the standards pass the resolution check criteria?				
5. Was the percent breakdown within QC limits?				
B. Continuing Calibration 1. Was the maximum %D within QC limits?				
2. Are compounds within retention time windows?				
3. Was the percent breakdown within QC limits?				
C. Sample Analysis After initial calibration, continuing calibration, and method blank criteria have been met: 1. Are sample name and other header information correct?				
2. Do surrogate % recoveries meet QC criteria?				
3. Were the runs checked for saturation?				
4. Are all hits confirmed if required?				
5. Are all compounds within linear range of calibration curve?				
6. Were all sample holding times met?				
7. Do reported results take into account dilutions, sample weights, and percent moistures?				
D. QC Samples 1. Are all Method Blank hits below the reporting limit?				

FIGURE 8.6-7
ITAS Data Review Check List
GC

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Level Review (✓)
D. QC Samples (continued) 4. Are MS/MSD and/or Duplicates (duplicate sample analyses) RPD within QC limits?				
5. Was second source check standard analyzed successfully?				
E. Other 1. Are all nonconformances included and noted?				
2. Are all required forms filled out?				
3. Correct methodology used?				
4. Transcriptions checked?				
5. Were all calculations checked at minimum frequency?				
6. Were all manual integrations checked by a second reviewer?				
7. Units checked?				

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

FIGURE 8.6-8
ITAS Data Review Check List
HPLC

Work Order Number(s):
Lab Sample Numbers or SDG:
Method/Test/Parameter:

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Level Review (✓)
A. Initial Calibration 1. Was the maximum %RSD within QC limits or was the correlation coefficient of the curve ≥ 0.995?				
2. Was each run checked for saturation?				
3. Was each run checked for coeluting compounds?				
B. Continuing Calibration 1. Was the maximum %D within QC limits?				
2. Was the run checked for saturation?				
3. Was each run checked for coeluting compounds?				
C. Method Blanks 1. Are all hits below the PQL?				
2. Was a Method Blank analyzed for each set of samples extracted by the same method on the same day?				
3. Do surrogate % recoveries meet QC criteria?				
4. Are peaks quantified against the appropriate standard?				
5. Was the run(s) checked for saturation?				
D. Sample Analysis After initial calibration, continuing calibration, and method blank criteria have been met: 1. Are sample name and other header information correct?				
2. Do surrogate % recoveries meet QC criteria?				
3. Were the runs checked for saturation?				
4. Are all hits confirmed?				
5. Were all sample holding times met?				

FIGURE 8.6-8
ITAS Data Review Check List
HPLC

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Level Review (✓)
E. QC Samples 1. Are matrix spike/matrix spike duplicate (MS/MSD) spike % recoveries within QC limits?				
2. Are Laboratory Control Sample (LCS) spike % recoveries within QC limits?				
3. Are MS/MSD and/or Duplicates (duplicate sample analyses) RPD within QC limits?				
F. Other 1. Are all nonconformances included and noted?				
2. Are all required forms filled out?				
3. Correct methodology used?				
4. Transcriptions checked?				
5. Were all calculations checked at minimum frequency?				
6. Were all manual integrations checked by a second reviewer?				
7. Units checked?				

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date:

FIGURE 8.6-9
ITAS Data Review Check List
DIOXINS/DIBENZOFURANS

Work Order Number(s):
Lab Sample Numbers or SDG:
Method/Test/Parameter:

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Level Review (✓)
A. Initial Calibration 1. Percent RSD within QC limits?				
2. Windowing solution analyzed and retention time windows determined?				
3. Were all signal/noise ratios met?				
4. Were all ion isotopic ratios within specifications?				
5. Was the 2,3,7,8-TCDD resolution met?				
B. Continuing Calibration 1. Continuing calibration standard results within QC limits?				
2. Were all signal/noise ratios met?				
3. Were all ion isotopic ratios within specifications?				
4. Was the window defining solution analyzed at the appropriate frequency?				
5. Were the proper types of continuing calibration check solutions run at the appropriate frequency?				
6. Was the 2,3,7,8-TCDD resolution met?				
C. Sample Analysis 1. Were sample holding times met?				
2. All analyte hits and NDs within set retention time windows?				
3. Are internal standard recoveries within QC limits?				
4. Was a minimum of 20% of the raw data recalculated?				
5. Were signal/noise ratios met for positive results?				
6. Were ion isotopic ratios met for positive results?				

FIGURE 8.6-9
ITAS Data Review Check List
DIOXINS/DIBENZOFURANS

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Level Review (✓)
D. QC Sample Analysis 1. All Method Blank results "None Detected"?				
2. Native spike recoveries of batch MS/MSD within QC limits?				
3. Native spike recoveries of batch LCS (or Blank Spike) within QC limits?				
4. Are internal standard recoveries within QC limits?				
5. Is the RPD of batch MS/MSD within QC limits?				
E. Other 1. Are all nonconformances included and noted?				
2. Are all required forms filled out?				
3. Correct methodology used?				
4. Transcriptions checked?				
5. Were all calculations checked at minimum frequency?				
6. Were all manual integrations checked by a second reviewer?				
7. Was % moisture and/or % lipids performed?				
8. Were sample results corrected for % moisture if requested by a client?				
9. Units checked?				

Comments on any "No" response:

_____

_____

_____

_____

_____

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

FIGURE 8.6-10
Example Certificate of Analysis
Page 1 of 2

IT INTERNATIONAL TECHNOLOGY CORPORATION

ANALYTICAL SERVICES

CERTIFICATE OF ANALYSIS

Client Name
Client Company
Client Address

Date:

Work Order No.

This is a Certificate of Analysis for the following samples:

Client Project ID:
Date Samples Received:
Number of Samples:

I. Introduction

This section describes general project information. It also includes the date the samples were received and provides a listing of the sample numbers. (Both client and laboratory numbers.)

Reviewed and Approved:

Project Manager

FIGURE 8.6-10
Example Certificate of Analysis
Page 2 of 2

IT ANALYTICAL SERVICES
304 DIRECTORS DRIVE
KNOXVILLE, TN
(615) 690-3211

Client Project ID ITAS Project No.:

II. Analytical Data

This section describes the sample preparation and analytical methodologies used to extract and analyze the samples. It also describes how the sample results are reported. Additionally, any project anomalies, nonconformances, variance, or any other type of unusual occurrences that were noted by the laboratory are documented here.

III. Quality Assurance/Quality Control

This section describes what type or level of QA/QC was followed. QA/QC requirements may be internal or client-specific. Any QA/QC data that exceeded the acceptance criteria will be noted here. Comments may also be included regarding how the QC sample results may have affected the data.

(The sample results follow on additional pages in tabular form.)

FIGURE 9.3-1
ITAS Nonconformance Memo
PAGE 1 of 2

LOG #: _-93-_____ page 1 of 2

ITAS-_____ Laboratory
LABORATORY NONCONFORMANCE MEMO (NCM)

PROJECT ID (Name/Number): _____
NCM INITIATED BY (Name/Date): _____
PARAMETER(S): _____
SAMPLE NUMBER(S) AFFECTED: _____

AREA:

☐ SHP/REC	☐ GC	☐ GEN CHEM	☐ BIOASSAY	☐ IH
☐ ORG EXT	☐ HPLC	☐ METALS	☐ RADIOCHEM	☐ DATA VERF
☐ INORG PREP	☐ GCMS	☐ GEO	☐ COUNTING	☐ REPORTING
☐ OTHER	☐ _____	☐ _____	☐ _____	☐ _____

NONCONFORMANCE (check appropriate item(s)):

1. ☐ Not enough sample received for proper analysis.
2. ☐ Holding time exceeded by _____ days due to:
   - 2.1 ☐ **CATEGORY I: Out of Laboratory Control**
     - ☐ Holding time expired at receipt.
   - 2.2 ☐ **CATEGORY II: Laboratory Dependent**
     - ☐ work backlog ☐ instrument failure
     - ☐ communication ☐ other (see #10)
   - 2.3 ☐ **CATEGORY III: Laboratory Reruns**
     - 2.3.1 ☐ *QA/QC:*
       - ☐ surrogates ☐ internal standards
       - ☐ spike recoveries ☐ blank contamination
     - 2.3.2 ☐ *CONFIRMATION:*
       - ☐ second column ☐ contamination check
       - ☐ other (see #10)
     - 2.3.3 ☐ *DILUTION:*
       - ☐ over calibration ☐ under calibration
       - ☐ other (see #10)
     - 2.3.4 ☐ *OTHER: (see #10)*
3. ☐ Sample lost during extraction/analysis: no re-prep or re-analysis possible.
4. ☐ QC data reported to client outside of:
   - ☐ method limits ☐ internal limits
   - ☐ QAPP limits ☐ contract limits
   - ☐ regulatory limits ☐ blank criteria
5. ☐ Incorrect procedure(s) used. (See #10)
6. ☐ Invalid instrument calibration. (See #10)
7. ☐ Incorrect/incomplete data reported to client. (See #10)
8. ☐ Reported detection limit(s) higher than:
   - ☐ method limits ☐ QAPP limits
   - ☐ contract limits ☐ other (see #10)

   Due to:
   - ☐ sample matrix ☐ insufficient sample
   - ☐ instrumentation ☐ other (see #10)
9. ☐ Other (specify): _____
10. ☐ Comments/Explanation: _____

NOTIFICATION (check appropriate item(s)):

1. ☐ Client notified by (name and date): _____
   - ☐ in writing ☐ by FAX
   - ☐ by phone ☐ Other (explain)
2. ☐ Client's name _____ and response:
   - ☐ process "as is" ☐ resample
   - ☐ on hold til ___ ☐ Other (explain)

PROJECT MANAGER (signature & date): _____

FURTHER ACTION REQUIRED, SEE PAGE 2 OF 2

FIGURE 9.3-1
ITAS Nonconformance Memo
Page 2 of 2

INTERNATIONAL TECHNOLOGY CORPORATION LOG # _-93-_____ page 2 of 2

CORRECTIVE ACTION

☐ ROOT CAUSE: INITIALS/DATE_____

☐ CORRECTIVE ACTION: INITIALS/DATE_____

☐ RESPONSIBILITY FOR PERFORMING CORRECTIVE ACTION ASSIGNED TO:

☐ ACTIONS TO PREVENT RECURRENCE: INITIALS/DATE_____

FIRST LEVEL SUPERVISOR: _____ DATE: _____

RESPONSIBLE MANAGER: _____ DATE: _____

QC REVIEW

☐ NONCONFORMANCE ☐ DEFICIENCY

☐ FURTHER ACTION REQUIRED: _____

ASSIGNED TO: _____

QC COORDINATOR: _____ DATE: _____

CORRECTIVE ACTION VERIFICATION

☐ VERIFIED ☐ CANNOT VERIFY (specify reason)

REASON: _____

NCM CLOSURE

QC COORDINATOR: _____ DATE _____

SIGNED ORIGINAL MUST BE RETAINED IN FILE: ☐ QUALITY/OPERATIONS FILE ☐ PROJECT FILE

FIGURE 9.3-2
Example Nonconformance Log

M	DATE INITIATED	PROJECT ID/ WORK ORDER NUMBER	LAB AREA	NO. OF SAMPLES AFFECTED	NC CATEGORY	CORRECTIVE ACTION TARGET DATE	CORRECTIVE ACTION COMPLETION DATE	N OR D *	CORRECTIVE ACTION VERIFICATION DATE/INITIALS
0001									
0002									
0003									
0004									
0005									
0006									
0007									
0008									
0009									
0010									
0011									
0012									
0013									
0014									
0015									
0016									
0017									
0018									
0019									
0020									

*N = nonconformance; D = deficiency (NOTE: column 10 need not be completed for a deficiency)

FIGURE 9.4-1
ITAS Client Satisfaction Survey Form
(Page 1 of 2)

IT QUALITY / CLIENT SATISFACTION SURVEY

CLIENT INFORMATION:	PROJECT INFORMATION:
Client Company _____ Client Contact _____ Title _____ Address _____ _____ _____ Phone _____	IT Project Number _____ Project Name _____ Project Size ($000) _____ Work Description _____ _____ _____ Project Manager _____ _____
IT INFORMATION: Date of Survey _____ IT ANALYTICAL SERVICES DIVISION IT Center Performing Service _____ IT Host Center Name / Number _____ IT Host Center Location _____	

INTRODUCTION

IT is committed to maintaining and continually improving the quality of the services and products we offer. We define quality as meeting client requirements. Therefore, the best measure of our performance is our clients' level of satisfaction.

We would like to ask you to help us by answering a few short questions that rate our performance on a scale of 1 to 5.
A rating of "1" indicates totally unsatisfactory performance, a "3" is acceptable, and a "5" means outstanding.

	Rating (1-5)	Weight	Score
1. On this scale of 1 to 5, how well does IT meet your requirements overall?		2.00	
2. How would you rate the timeliness of our work?		2.00	
3. How well have we met your requirements in adhering to and completing the required scope of work?		2.00	
4. How well did we do in meeting your schedule requirements?		2.00	
5. How do you rate the responsiveness of our staff?		2.00	
6. How do you rate the professional competence of our staff?		2.00	
7 What is your assessment of how well we have communicated with you during the course of our work?		2.00	

FIGURE 9.4-1
ITAS Client Satisfaction Survey Form
(Page 2 of 2)

IT QUALITY / CLIENT SATISFACTION SURVEY p.2)

	Rating (1-5)	Weight	Score
8. How well does IT's management of our services meet your requirements?		2.00	
9. How well do our status and progress reports meet your requirements?		2.00	
10. How well do our invoices and billing procedures meet your requirements?		2.00	
11. How do you rate the consistency of our performance?		2.00	
12. How would you score the completeness of our services?		2.00	
13. How do you rate the overall value of the services and products provided to you by IT?		2.00	
TOTAL SCORE:			
AVERAGE SCORE:			
PERCENTAGE:	Total Possible:	130	

CLOSING

Thank you very much for your time. We appreciate your willingness to help us. Any concerns you may have raised will be addressed promptly by the IT Project Manager for your project.

There is one final question: What changes would you recommend that IT make to improve its service to you?

Again, Thank you. Survey Conducted by: _____

FOLLOWUP ACTION:	**Distribution:**
What and How:	Project Manager _____ Lab/FAS Director _____ Div. Dir./VP _____ VP QHS _____ Div. Dir QA/QC _____ Regional Director _____ Other _____
Who: Date:	
Reviewer _____	
Signature Date	

FIGURE 9.5-1
Example Outline Of QA/QCC Monthly Report to Management

I. Audits
   - A. Internal Surveillances
   - B. External Audits
   - C. Subcontractor Audits

II. Certifications
   - A. Pending
   - B. Received

III. Performance Evaluation Samples
   - A. In-House
   - B. Pending
   - C. Received (score)

IV. Holding Time Violations (HTV)
   - A. Total Holding Time Violations
   - B. Category I - Out of Laboratory Control
   - C. Category II - Laboratory Dependent
   - D. Category III - Laboratory Reruns

V. QAPjPs
   - A. Reviewed
   - B. Received

VI. Training
   - A. In-House
   - B. External

VII. Nonconformance Summary and Resolutions

VIII. QC Data/Control Chart Summary

IX. Standard Operating Procedures

X. Client Satisfaction Surveys

XI. Miscellaneous

TABLE 2.1-1
ITAS QUALITY ASSURANCE MANAGEMENT PLAN REQUIREMENTS MATRIX

ITAS QAMP (Rev.2) & OS QAMP (Rev.0)	ITAS QAM (REV 1)	ANSI/ASQC E4-19xx	QAMS 005/80	NQA-1	5700.6C	ANSI N13.30	ANSI/ASQC Q91-1987 (ISO-9001)
Management Commitment and Organization	2 Laboratory Organization	A1 Management Commitment and Organization	5.04 Project Organization and Responsibility	1 Organization	N/A	1.1 Introduction 1.2 Purpose 1.3 Scope	4.1 Management Responsibility
Quality Assurance Program Description	1 Introduction 3 Standard Laboratory Practice	A2 Quality Assurance Program Description	5.03 Project Description	2 Quality Assurance Program	1 Program	2.1 Special Word Usage 2.2 Specific Terms 5.1 Quality Assurance 5.2 Quality Control	4.2 Quality System
Associate Training and Qualification	16 Training	A3 Personnel Training and Qualification	N/A	2 Quality Assurance Program	2 Personnel Training and Qualification	3.2 Personnel Preparation	4.18 Training
Procurement of Items and Services	4 Material Procurement and Control	A5 Procurement of Items and Services	N/A	4 Procurement Document Control 7 Control of Purchased Items and Services	7 Procurement	N/A	4.6 Purchasing

TABLE 2.1-1
ITAS QUALITY ASSURANCE MANAGEMENT PLAN REQUIREMENTS MATRIX

ITAS QAMP (Rev.2) & OS QAMP (Rev.0)	ITAS QAM (REV 1)	ANSI/ASQC E4-19xx	QAMS 005/80	NQA-1	5700.6C	ANSI N13.30	ANSI/ASQC Q91-1987 (ISO-9001)
Document Control and Records	1 Introduction 12 Records Management	A6 Document Control and Records	5.01 Title Page 5.02 Table of Contents	6 Document Control 17 Quality Assurance Records	4 Documents and Records	3.6 Direct Bioassay - Record Retention 4.5 Indirect Bioassay - Record Retention	4.5 Document Control 4.16 Quality Records
Use of Computer Hardware and Software	10 Data Verification	A7 Use of Computer Hardware and Software	N/A	3 Design Control 11 Test Control	N/A	N/A	ISO 9000 3
Work Processes and Operations	9 Analytical Procedures 14 QA/QC Audits	A8 Work Processes and Operations B1 Planning and Scoping B2 Design of Data Collection Operations	N/A	1 Organization 5 Instructions, Procedures, and Drawings 10 Inspection 14 Inspection, Test and Operating Status	5 Work Processes 6 Design 8 Inspection and Acceptance Testing	3.1 Facility Criteria	4.9 Process Control

TABLE 2.1-1
ITAS QUALITY ASSURANCE MANAGEMENT PLAN REQUIREMENTS MATRIX

ITAS QAMP (Rev.2) & OS QAMP (Rev.0)	ITAS QAM (REV 1)	ANSI/ASQC E4-19xx	QAMS 005/80	NQA-1	5700.6C	ANSI N13.30	ANSI/ASQC Q91-1987 (ISO-9001)
Data Collection and Production Operations	5 Sample Receipt and Initiation of Testing	B1 Planning and Scoping	5.05 QA Objectives for Measurement Data	2 Quality Assurance Program	1 Program	3.1 Facility Criteria	4.8 Product Identification and Traceability
	6 Calibration Practices	B2 Design of Data Collection Operations	5.06 Sampling Procedures	3 Design Control	6 Design	3.4 Direct Bioassay - Performance Criteria for Service Laboratories	4.11 Inspection, Measuring, and Test Equipment
	7 Preventive Maintenance	B3 Implementation of Planned Operations	5.07 Sample Custody	5 Instructions, Procedures, and Drawings	8 Inspection and Acceptance Testing	3.5 Direct Bioassay - Reporting Results	
	8 Analysis of Quality Control Samples		5.08 Calibration Procedures and Frequency	8 Identification and Control of Items		4.1 Indirect Bioassay - Responsibilities of the Service Laboratory Customer	
	9 Analytical Procedures		5.09 Analytical Procedures	9 Control of Processes		4.2 Indirect Bioassay - Analytical Methodology	
	10 Data Verification		5.11 Internal Quality Control Checks	11 Test Control		4.3 Indirect Bioassay - Performance Criteria for Service Laboratories	
			5.13 Preventive Maintenance	12 Control of Measuring and Test Equipment		5.2 Quality Control	
				13 Handling, Storage, and Shipping			

TABLE 2.1-1
ITAS QUALITY ASSURANCE MANAGEMENT PLAN REQUIREMENTS MATRIX

TAS QAMP Rev.2) & OS AMP (Rev.0)	ITAS QAM (REV 1)	ANSI/ASQC E4-19xx	QAMS 005/80	NQA-1	5700.6C	ANSI N13.30	ANSI/ASQC Q91-1987 (ISO-9001)
Quality Assessment and Response	13 Nonconformance and Corrective Action	A4 Management Assessment	5.10 Data Reduction, Validation, and Reporting	2 Quality Assurance Program	3 Quality Improvement	3.3 Direct Bioassay-Interpretation of Measurements	4.10 Inspection and Testing
	14 Quality Assurance/Quality Control Audits	A9 Quality Improvement	5.12 Performance and System Audits	13 Handling, Storage, and Shipping	9 Management Assessment	3.5 Direct Bioassay-Reporting Results	4.13 Control of Nonconforming Products
	15 Quality Reports to Management	B4 Assessment of Data Usability	5.14 Specific Routine Procedures Used to Assess Data Precision, Accuracy, and Completeness	15 Control of Nonconforming Items	10 Independent Assessment	4.4 Indirect Bioassay-Reporting Results	4.14 Corrective Action
		B5 Quality Assessment and Response	5.15 Corrective Action	16 Corrective Action		6.1 Direct Bioassay Measurements	4.17 Internal Quality Audits
			5.16 Quality Assurance Reports to Management	18 Audits		6.2 Indirect Bioassay Measurements	4.20 Statistical Techniques

y Management and Quality Assurance Standards, ISO 9000, Part 3, "Guidelines for the application of ISO 9001 to the development, supply and maintenance of software".

TABLE 2.3-1
ITAS Quality Assurance Documents

Document	Purpose	Required Approval
ITAS Quality Assurance Management Plan (QAMP)	• Provides ITAS Quality Assurance Policy • Is applicable to and provides direction for all ITAS laboratories • Covers administrative and technical aspects of QA/QC • Has precedence over all other ITAS quality related documents	• Chief Executive Officer (CEO), IT Corporation • Vice President, Quality and Health Services, IT Corporation • Vice President, IT Analytical Services • Division Director, Quality Assurance/Quality Control
Operation-Specific Quality Assurance Management Plan (OS QAMP)	• Describes the local implementation of the policies in the ITAS Quality Assurance Management Plan • Meets or exceeds all requirements of the ITAS QAMP • Describes services and quality requirements common to ITAS operations	• Vice President, Analytical Services, IT Corporation • Division Director, Quality Assurance/Quality Control • Vice President, Quality and Health Services • Division Operations Director • Operating Unit Director
OS QAMP Operation-Specific Appendices	• Describes services and quality requirements unique to each ITAS operation	• Laboratory QA/QCC • Technical Specialist • Operating Unit Director • Division Director, QA/QC • Division Technical Director
Manuals of Practice (MOP)	• Provides in-depth technical discussions of a specific topic • May be a collection of standard operating procedures (SOPs).	• Vice President, IT Analytical Services • Division Director, Quality Assurance/Quality Control

TABLE 2.3-1
ITAS Quality Assurance Documents
(Continued)

Document	Purpose	Required Approval
Division Standard Operating Procedures (SOP) (formerly ITAS Systems Procedures)	• Common to all ITAS operations and standard across the network • Provides detailed instructions describing how to perform a specific operation or task • Defines responsibilities as related to the operation or task • Defines quality requirements as related to the operation or task.	• ITAS Technical Specialist • Division Director, Quality Assurance/Quality Control • Division Director, Health and Safety • ITAS Standardization Committee Member
Laboratory-Specific Standard Operating Procedures (SOP)	• Provides detailed instructions describing how to perform a specific operation or task • Defines responsibilities as related to the operation or task • Defines quality requirements as related to the operation or task.	• Division Director, Quality Assurance/Quality Control • Division Director, Health and Safety • Operating Unit Director • Laboratory Technical Director • Laboratory QA/QCC
Project Specific Manuals, Quality Assurance Project Plans (QAPjPs), or Quality Assurance Program Plans (QAPPs)[1]	• Defines project requirements • Defines the organizational structure for a project and lines of communication between the parties • Defines contractual requirements as pertains to sample analysis • Defines regulatory requirements • Takes precedence over conventional ITAS QA practices for the project	• Operating Unit Director • Laboratory QA/QCC • Laboratory Technical Director • Laboratory Health and Safety Coordinator • Project Manager • Client Representative

TABLE 2.3-1
ITAS Quality Assurance Documents
(Continued)

Document	Purpose	Required Approval
Quality Assurance Summary (QAS) (Operation-specific document)	• Defines specific QA/QC requirements • Defines reporting requirements • Defines any client-specific requirement that varies from normal operation	• Approval varies within the operations

(1) Listed approvals required apply only to these documents when generated by ITAS.

TABLE 8.2-1
Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2], [7]	CLP[3],[4]	Other
Acidity	Water	250 ml plastic or glass, Cool, 4°C, 14 days	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Alkalinity	Water	250 ml plastic or glass, Cool, 4°C, 14 days	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Ammonia	Water	500 ml plastic or glass, Cool, 4°C H_2SO_4 to pH < 2, 28 days	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Biochemical Oxygen Demand (BOD)	Water	1000 ml plastic or glass, Cool, 4°C 48 hours	Not Applicable	Not Applicable	200 ml, no preservative required, 48 hours
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable

Table 8.2-1
Inorganic Sample Containers, Preservations, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2], [7]	CLP[3],[4]	Other
Biochemical Oxygen Demand (BOD) (continued)	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Bromide	Water	250 ml plastic or glass, No preservative required, 28 days	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Chemical Oxygen Demand (COD)	Water	250 ml glass or plastic, Cool, 4°C, H_2SO_4 to pH < 2, 28 days	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	2 or 4 oz glass, No preservative
Chloride	Water	250 ml plastic or glass, No preservative required, 28 days	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable

Table 8.2-1
Inorganic Sample Containers, Preservations, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2], [7]	CLP[3],[14]	Other
Residual Chlorine	Water	250 ml plastic or glass, No preservative required, analyze immediately	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not applicable
Chromium (Cr^{+6})	Water	200 ml plastic or glass, Cool, 4°C, 24 hours	250 ml plastic or glass, Cool, 4°C, 24 hours	Not Applicable	Not Applicable
	Liquid	200 ml plastic or glass, Cool, 4°C, 24 hours	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Color	Water	250 ml plastic or glass, Cool, 4°C, 48 hours	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Conductivity	Water	250 ml plastic or glass, Cool, 4°C, 28 days	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable

Table 8.2-1
Inorganic Sample Containers, Preservations, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2], [7]	CLP[3],[4]	Other
Conductivity (continued)	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cyanide (Amenable)	Water	1 liter plastic or glass, NaOH to pH > 12 0.6g ascorbic acid[6] Cool, 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours	1 liter plastic or glass NaOH to pH > 12 0.6g ascorbic acid[6] Cool, 4°C, 14 days	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	1 liter HDPE[5] NaOH to pH > 12 0.6g ascorbic acid[6] Cool, 4°C
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cyanide (Free)	Water	Not Applicable	Not Applicable	Not Applicable	1 liter HDPE[5], NaOH to pH > 12 0.6g ascorbic acid[6] Cool, 4°C, 14 days
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	8 oz or 16 oz glass teflon lined lid, Cool, 4°C, 14 days

Table 8.2-1
Inorganic Sample Containers, Preservations, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2], [7]	CLP[3],[4]	Other
Cyanide (Total)	Water	1 liter plastic or glass, NaOH to pH > 12, 0.6g ascorbic acid[6] Cool, 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours	1 liter plastic or glass, NaOH to pH > 12 0.6g ascorbic acid[6] Cool,4°C, 14 days	1 liter HDPE[5], NaOH to pH > 12 Cool, 4°C, 12 days	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	TCLP Leachate: 14 days Analysis: 14 days from end of TCLP leaching not to exceed 28 days total	Not Applicable	1 liter HDPE[5] NaOH to pH > 12 0.6g ascorbic acid[6] Cool, 4°C
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	8 or 16 oz glass teflon-lined lids, Cool, 4°C, 14 days	8 or 16 oz glass teflon-lined lids, Cool, 4°C, 12 days	Not Applicable
Flashpoint (Ignitability)	Water	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	No requirements: 250 ml amber glass, Cool, 4°C is recommended	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Fluoride	Water	500 ml plastic, No preservation required, 28 days	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste,	Not Applicable	Not Applicable	Not Applicable	Not Applicable

INTERNATIONAL TECHNOLOGY CORPORATION

Procedure Change No.:

OSOAMP-04

IT ANALYTICAL SERVICES DIVISION PROCEDURE/DOCUMENT CHANGE

PROCEDURE/DOCUMENT NUMBER: N/A			
PROCEDURE/DOCUMENT TITLE: ITAS Operation-Specific Quality Assurance Management Plan (OS QAMP) (Revision 0, September 1, 1993)			
PROCEDURE/DOCUMENT SECTION(S) AFFECTED BY CHANGE: Table 8.2-1, "Inorganic Sample Containers, Preservatives, and Holding Times", page 133.			
REASON FOR ADDITION OR CHANGE: Addition of Hydrazine (colorimetric) to the table.			
CHANGE EFFECTIVE FROM:	02/18/94	TO:	Ongoing
SAMPLES OR PROJECTS AFFECTED: N/A			
CHANGE: Add the attached table containing the requirements for hydrazine in front of page 133.			
SUBMITTED BY/DATE: Nasreen DeRubeis 02/07/94			
APPROVED BY:			
James K. Lahlè 2-23-94		TECHNICAL SPECIALIST/DATE	
Lian E. Ruppla 2/16/94		ITAS DIRECTOR, HEALTH & SAFETY/DATE	
John Hall 2/17/94		ITAS DIRECTOR, QA/QC/DATE	

ADDITION TO:

TABLE 8.2-1

Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2, 7]	CLP[3],[6]	Other
Hydrazine Colorimetric	Water	Not Applicable	Not Applicable	Not Applicable	100 ml amber glass, HCl to pH < 2, Cool, 4°C, 28 days
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	100 g amber glass, Cool, 4°C, 28 days

Table 8.2-1
Inorganic Sample Containers, Preservations, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2], [7]	CLP[3],[4]	Other
Fluoride (continued)	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Hardness (Total)	Water	250 ml plastic or glass, HNO_3 or H_2SO_4 to pH < 2, 6 months	Not Applicable	Not Applicable	1 liter glass or polyethylene, pH ≤ 2 with HNO_3, 6 months
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Iodide	Water	100 ml plastic or glass, Cool, 4°C, 24 hours	Not Applicable	Not Applicable	100 ml HDPE[5], Cool, 4°C, 24 hours
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Methylene Blue Active Substances (MBAS) (Surfactant)	Water	250 ml plastic or glass, Cool, 4°C, 48 hours	Not Applicable	Not Applicable	250 ml HDPE[5], Cool, 4°C, 48 hours
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable

Table 8.2-1
Inorganic Sample Containers, Preservations, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2], [7]	CLP[3],[4]	Other
Nitrogen, Nitrate-Nitrite	Water	250 ml plastic or glass, Cool, 4°C, H_2SO_4 to pH < 2, Nitrate - 14 days preserved, 48 hours unpreserved, Nitrite - 48 hours, Nitrate + Nitrite - 28 days preserved	Cool, 4°C, plastic or glass, 48 hours	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Odor	Water	200 mL glass only, Cool, 4°C, 24 hours	Not Applicable	Not Applicable	200 mL glass only, Cool, 4°C, 24 hours
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Oil and Grease	Water	1 liter glass, Cool, 4°C HCl or H_2SO_4 to pH < 2, 28 days	1 liter glass, Cool, 4°C H_2SO_4 to pH < 2, 28 days	Not Applicable	1 liter amber glass, Cool, 4°C H_2SO_4 to pH < 2, 28 days
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	8 oz amber glass with teflon-lined lid, Cool, 4°C, 28 days	Not Applicable	1 liter amber glass, Cool, 4°C H_2SO_4 to pH < 2, 28 days (Not applicable to Sludge, solid, or sediment)

Table 8.2-1
Inorganic Sample Containers, Preservations, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2], [7]	CLP[3],[4]	Other
Oil and Grease (continued)	Soil	Not Applicable	8 oz amber glass with teflon-lined lid, Cool, 4°C, 28 days	Not Applicable	Not Applicable
Ortho-phosphorous	Water	100 ml plastic or glass, Filter on site Cool 4°C, 48 hours	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
pH	Water	100 ml plastic or glass Analyze immediately. **This test should be performed in the field**	100 ml plastic or glass, Analyze immediately. **This test should be performed in the field**	Method CLP, Analyze as close to extraction as possible	Not Applicable
	Liquid	Not Applicable	100 ml glass, Cool, 4°C, Analyze immediately. **This test should be performed in the field**	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	This test is performed in the laboratory	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	4 oz. glass or plastic, Cool, 4°C, Analyze as soon as possible.	Not Applicable	Not Applicable
Phenolics	Water	500 ml glass, Cool, 4°C H_2SO_4 to pH < 2, 28 days	1 liter glass recommended, Cool, 4°C H_2SO_4 to pH < 2, 28 days	Not Applicable	Not Applicable

Table 8.2-1
Inorganic Sample Containers, Preservations, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2], [7]	CLP[3],[4]	Other
Phenolics (continued)	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Phosphorus (Total)	Water	100 ml plastic or glass, Cool, 4°C H_2SO_4 to pH < 2, 28 days	Not Applicable	Not Applicable	100 ml HDPE[5], Cool, 4°C H_2SO_4 to pH < 2, 28 days
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Reactivity (Cyanide and Sulfide)	Water	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	10 oz glass amber, Cool, 4°C, no headspace, analyze as soon as possible	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Settleable Solids	Water	1000 ml plastic or glass, Cool, 4°C, 48 hours	Not Applicable	Not Applicable	1 liter HDPE[5], Cool, 4°C, 24 hours
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Silica, Dissolved	Water	Plastic only, 100 mL, Cool, 4°C,	Not Applicable	Not Applicable	Plastic only, 100 mL, Cool, 4°C,

Table 8.2-1
Inorganic Sample Containers, Preservations, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2], [7]	CLP[3],[4]	Other
Silica, Dissolved (continued)	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Sulfate (SO_4)	Water	100 ml plastic or glass, Cool, 4°C, 28 days	100 ml plastic or glass Cool, 4°C, 28 days	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	100 ml plastic or glass Cool, 4°C, 28 days Not applicable to sludge, solid, and sediment.	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Sulfide	Water	500 ml plastic or glass, Cool, 4°C, Add 2 ml zinc acetate plus NaOH to pH > 9, 7 days	500 ml HDPE[9], Cool, 4°C Add 2 ml zinc acetate plus NaOH to pH > 9, 7 days	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Fill the surface of the solid with 2N zinc acetate until moistened. Cool, 4°C, store headspace-free	Not Applicable	Not Applicable
	Soil	Not Applicable	Fill the surface of the solid with 2N zinc acetate until moistened. Cool, 4°C, store headspace-free	Not Applicable	Not Applicable

Table 8.2-1
Inorganic Sample Containers, Preservations, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2], [7]	CLP[3],[4]	Other
Sulfite (SO_3)	Water	100 ml plastic or glass, No preservative required, analyze immediately	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Temperature	Water	1 liter plastic or glass, analyze immediately in the field	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Total Dissolved Solids	Water	250 ml plastic or glass, Cool, 4°C, 7 days	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Total Kjeldahl Nitrogen (TKN)	Water	500 ml plastic or glass, Cool, 4°C H_2SO_4 to pH < 2, 28 days	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	[illegible]	Not Applicable	Not Applicable	Not Applicable	Not Applicable

Table 8.2-1
Inorganic Sample Containers, Preservations, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2], [7]	CLP[3],[4]	Other
Total Organic Carbon (TOC)	Water	100 ml plastic or glass, H_2SO_4 or HCl to pH < 2 Cool, 4°C, 28 days	100 ml plastic or glass, H_2SO_4 or HCl to pH < 2 Cool, 4°C, 28 days	Not Applicable	100 ml HDPE[5], H_2SO_4 or HCl to pH < 2, Cool, 4°C, 28 days
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Total Organic Halides (TOX)	Water	Not Applicable	500 ml glass amber teflon-lined lid, Cool, 4°C H_2SO_4 to pH < 2, no headspace, 28 days	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
TPH	Water	1 liter glass, H_2SO_4 to pH<2, 28 days	1 liter glass, H_2SO_4 to pH<2, 28 days	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	4 or 8 oz glass widemouth with Teflon-lined lid, Cool, 4°C, 28 days	Not Applicable	Not Applicable
	Soil	Not Applicable	4 or 8 oz glass widemouth with Teflon-lined lid, Cool, 4°C, 28 days	Not Applicable	250 ml glass, no preservative required, 28 days
Total Solids	Water	250 ml plastic or	Not Applicable	Not Applicable	Not Applicable

Table 8.2-1
Inorganic Sample Containers, Preservations, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2], [7]	CLP[3],[4]	Other
Total Solids (continued)	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Total Suspended Solids	Water	250 ml plastic or glass, Cool, 4°C, 7 days	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Total Volatile Solids	Water	250 ml plastic or glass, Cool, 4°C, 7 days	Not Applicable	Not Applicable	250 ml, HDPE[m], Cool, 4°C, 7 days
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Turbidity	Water	250 ml plastic or glass, Cool, 4°C, 48 hours	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable

Table 8.2-1
Inorganic Sample Containers, Preservations, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2], [7]	CLP[3],[4]	Other
ICAP, GFAA, and Flame AA (excludes mercury)	Water	1 liter glass or polyethylene container, HNO_3 to pH ≤ 2, 6 months	1 liter glass or polyethylene container, pH ≤ 2 with HNO_3, 6 months	1 liter glass or polyethylene, Cool, 4°C, pH ≤ 2 with HNO_3, 180 days	Not Applicable
	Liquid	Not Applicable	No preservative required, 6 months	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	1 liter plastic or glass (glass only if organics are to be tested from the same bottle). HNO_3 to pH < 2, TCLP Leaching: 180 days from field collection to TCLP extraction. Analysis: 180 days from extraction to analysis (total elapsed time = 360 days)	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	1 liter glass or polyethylene container, 6 months	8 or 16 oz glass or polyethylene container Cool, 4°C, 6 months	Not Applicable	Not Applicable
	Soil	Not Applicable	8 or 16 oz glass or polyethylene container, Cool, 4°C, 6 months	8 or 16 oz glass or polyethylene container, Cool, 4°C, 180 days	Not Applicable
	Air	Not Applicable	Not Applicable	Not Applicable	Hi-Vols - store in humidity controlled environment, no holding time requirement. IH - no holding time requirement
Mercury (CVAA)	Water	1 liter glass or polyethylene container, Cool, 4°C, HNO_3 to pH ≤ 2, 28 days	1 liter glass or polyethylene container, HNO_3 to pH ≤ 2, 13 days plastic, 38 days glass	1 liter glass or polyethylene container, Cool, 4°C, HNO_3 to pH ≤ 2, 26 days	Not Applicable
	Liquid	Not Applicable	No preservative	Not Applicable	Not Applicable

Table 8.2-1
Inorganic Sample Containers, Preservations, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2], [7]	CLP[3],[4]	Other
Mercury (CVAA) (continued)	TCLP Leachate	Not Applicable	1 liter HDPE[5], HNO_3 to pH < 2, TCLP Leaching: 28 days from field collection to TCLP extraction. Analysis: 28 days from extraction to analysis (total elapsed time = 56 days.	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	8 or 16 oz glass or polyethylene container, Cool, 4°C, 28 days	8 or 16 oz glass or polyethylene container, Cool, 4°C, 28 days	1 liter glass or polyethylene container, Cool, 4°C, 26 days	Not Applicable
	Soil	8 or 16 oz glass or polyethylene container, Cool, 4°C, 28 days	8 or 16 oz glass or polyethylene container, Cool, 4°C, 28 days	1 liter glass or polyethylene container, Cool, 4°C, 26 days	Not Applicable
	Air	Not Applicable	Not Applicable	Not Applicable	Hi-Vols - store in humidity controlled environment, no holding time requirement IH - no holding time requirement

(1) National Pollutant Discharge Elimination System
(2) Resource Conservation and Recovery Act (Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd edition, Final Update I, July 1992
(3) Contract Laboratory Program
(4) Holding times are calculated from verified time of sample receipt
(5) High density Polyethylene
(6) Should be used only in the presence of chlorine
(7) Holding times are calculated from date of collection

TABLE 8.2-2
Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[20,47]	CLP 88 & 90[46, 48]	Other
Aromatic Volatiles	Water[44]	40 ml glass VOA vial (in duplicate) with teflon-lined septa without headspace, Cool 4°C. Add sodium thiosulfate if residual chlorine, 7 days with pH > 2 14 days with pH ≤ 2	40 ml glass VOA vial (in duplicate) with teflon-lined septa without headspace, Cool 4°C, Add sodium thiosulfate if residual chlorine, HCl or H_2SO_4 or solid $NaHSO_4$ to pH ≤ 2, 14 days with pH ≤ 2	Not Applicable	40 ml glass VOA vial with teflon-lined septa in duplicate without headspace, Cool 4°C, HCl to pH ≤ 2,
	Liquid	Not Applicable	40 ml glass VOA with teflon-lined lid, Cool 4°C, 14 days	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	4 or 8 oz glass with teflon-lined lid, Cool, 4°C, TCLP Leachate: 14 days, Analysis: 14 days from end of TCLP leaching (not to exceed 28 days total)	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	4 or 8 oz glass with teflon-lined lid, Cool 4°C, 14 days	Not Applicable	Not Applicable
	Soil	Not Applicable	4 or 8 oz glass with teflon-lined lid, Cool, 4°C, 14 days	Not Applicable	Not Applicable
	Air	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Dioxins/ Dibenzofurans	Water[45]	1 liter glass amber with teflon-lined lid, Cool, 4°C, Extract, 7 days Analysis, 40 days	1 liter glass amber with teflon-lined lid, Cool, 4°C, Extract, 30 days Analysis, 45 days from date of collection	Not Applicable	Not Applicable
	Liquid	Not Applicable	100 ml glass amber with teflon-lined lid, Cool, 4°C, Extract, 30 days Analysis 45 days from date of collection	Not Applicable	Not Applicable

Table 8.2-2
Organic Sample Containers, Preservatives, and Holding Times (continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2],[3]	CLP 88 & 90[4],[5]	Other
Dioxins/ Dibenzofurans (continued)	TCLP Leachate	Not Applicable	1 liter glass amber with teflon-lined lid. Cool, 4°C. Extract, 30 days Analysis 45 days from date of collection	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	8 or 16 oz glass amber wide mouth with teflon-lined lid. Cool 4°C. Extract, 30 days Analysis 45 days from date of collection	Not Applicable	Not Applicable
	Soil	Not Applicable	8 or 16 oz glass amber wide mouth with teflon-lined lid. Cool 4°C. Extract, 30 days Analysis 45 days from date of collection	Not Applicable	Not Applicable
	Air	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Formaldehyde	Water[11]	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Air	Not Applicable	Not Applicable	Not Applicable	Store at 4°C. Analysis, 30 days
Herbicides	Water[10]	1 liter glass amber with teflon-lined lid. Sodium thiosulfate of ascorbic acid if residual chlorine present. Cool, 4°C. Extract, 7 days Analysis, 40 days after extraction	1 liter glass amber with teflon-lined lid. Sodium thiosulfate if residual chlorine present. Cool, 4°C. Extract, 7 days Analysis, 40 days after extraction	Not Applicable	1 liter glass Cool, 4°C. Extract, 14 days. Analysis, 28 days
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable

Table 8.2-2
Organic Sample Containers, Preservatives, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2],[3]	CLP 88 & 90[4],[5]	Other
Herbicides (continued)	TCLP Leachate	Not Applicable	4 or 8 oz glass widemouth with teflon-lined lid, Cool, 4°C, TCLP Leachate, 14 days, Extract, 7 days from end of TCLP leaching (not to exceed 21 days total), Analysis, 40 days after extraction	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment		4 or 8 oz glass widemouth with teflon-lined lid, Cool, 4°C, Extract, 14 days, Analysis, 40 days after extraction	Not Applicable	Not Applicable
	Soil	Not Applicable	4 or 8 oz glass widemouth with teflon-lined lid, Cool, 4°C, Extract, 14 days Analysis, 40 days after extraction	Not Applicable	Not Applicable
	Air	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Nitroaromatics	Water[11]	Not Applicable	100 ml glass with teflon-lined cap, Cool, 4°C	Not Applicable	1 liter glass amber with teflon-lined lid, Cool, 4°C, Extract, 7 days Analysis, 40 days
	Liquid	Not Applicable	Not Applicable	Not Applicable	100 ml glass amber with teflon-lined lid, Cool, 4°C, Extract, 14 days Analysis, 40 days
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	4 or 8 oz glass amber widemouth with teflon-lined lid Room temperature or cooler	Not Applicable	8 or 16 oz glass amber wide mouth with teflon-lined lid, Cool, 4°C, Extract, 14 days Analysis, 40 days

Table 8.2-2
Organic Sample Containers, Preservatives, and Holding Times (continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2,3]	CLP 88 & 90[4, 5]	Other
Nitroaromatics (continued)	Soil	Not Applicable	4 or 8 oz glass amber widemouth with teflon-lined lid Room temperature or cooler	Not Applicable	8 or 16 oz glass amber wide mouth with teflon-lined lid, Cool, 4°C, Extract, 14 days Analysis, 40 days
	Air	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Non-Methane Organic Compounds (NMOC)	Water[11]	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Air	Not Applicable	Not Applicable	Not Applicable	Summa Cannister, No preservation or holding time required
PAH	Water[11]	1 liter glass amber with teflon-lined lid. Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present. Cool, 4°C, Extract, 7 days Analysis, 40 days after extraction	1 liter glass amber with teflon-lined lid, If residual chlorine present, add sodium thiosulfate. Cool, 4°C, Extract, 7 days Analysis, 40 days after extraction	Not Applicable	Not Applicable
	Liquid	Not Applicable	100 ml glass amber with teflon-lined lid, No preservative required, Extract, 14 days Analysis, 40 days after extraction	Not Applicable	Not Applicable

Table 8.2-2
Organic Sample Containers, Preservatives, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2,3]	CLP 88 & 90[4,5]	Other
PAH (continued)	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	4 or 8 oz widemouth glass with teflon-lined lid. Cool, 4°C. TCLP Leaching: 14 days from field collection to TCLP extraction. Extract 7 days from the end of TCLP leaching (not to exceed 14 days total). Analysis, 40 days after extraction
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	4 or 8 oz glass widemouth with teflon-lined lid. Cool, 4°C. Extract, 14 days Analysis, 40 days after extraction	Not Applicable	Not Applicable
	Soil	Not Applicable	4 or 8 oz glass widemouth with teflon-lined lid. Cool, 4°C. Extract, 14 days Analysis, 40 days after extraction	Not Applicable	Not Applicable
	Air	Not Applicable	Not Applicable	Not Applicable	PUF/XAD-2. Cool, 4°C. Extract within 7 days of collection. Analyze within 40 days from extraction

Table 8.2-2
Organic Sample Containers, Preservatives, and Holding Times (continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2,3]	CLP 88 & 90[4, 5]	Other
Pesticides/ PCBs	Water[6]	1 liter glass with teflon-lined lid. Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present and aldrin is being determined. Cool, 4°C. Extract, 7 days Analysis, 40 days after extraction	1 liter amber with teflon-lined lid. If residual chlorine present, add sodium thiosulfate. Cool, 4°C. Extract, 7 days Analysis, 40 days after extraction	1 liter glass amber with teflon-lined lid. Cool, 4°C. Extract within 5 days of sample receipt Analysis, 40 days after extraction	1 liter glass (in duplicate) with teflon-lined septa. Cool, 4°C. Extract, 14 days. Analysis, 30 days --- 1 liter glass amber with teflon-lined lid. Cool, 4°C. Extract, 30 days Analysis, 45 days from date of collection --- 2 X 40 ml glass amber with teflon-lined lid. Cool, 4°C. Extract 7 days Analyze 14 days from date of collection
	Liquid	Not Applicable	100 ml glass with teflon-lined lid. No preservative required Extract, 14 days Analysis, 40 days after extraction	Not Applicable	100 ml glass amber with teflon-lined lid. Cool, 4°C. Extract, 30 days Analysis, 45 days from date of collection
	TCLP Leachate	Not Applicable	4 or 8 oz widemouth glass with teflon-lined lid. Cool, 4°C. TCLP Leaching: 14 days from field collection to TCLP extraction. Extract 7 days from the end of TCLP leaching (not to exceed 21 days total). Analysis, 40 days after extraction	Not Applicable	1 liter glass amber with teflon-lined lid. Cool, 4°C. Extract, 30 days Analysis, 45 days from date of collection

Table 8.2-2
Organic Sample Containers, Preservatives, and Holding Times (continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2,3]	CLP 88 & 90[4,5]	Other
Pesticides/ PCBs (continued)	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	4 or 8 oz glass widemouth with teflon-lined lid, Cool, 4°C, Extract, 14 days Analysis, 40 days after extraction	Not Applicable	8 or 16 oz glass amber wide mouth with teflon-lined lid, Cool 4°C, Extract, 30 days Analysis, 45 days from date of collection
	Soil	Not Applicable	4 or 8 oz glass widemouth with teflon-lined lid, Cool, 4°C, Extract, 14 days Analysis, 40 days after extraction	4 or 8 oz glass widemouth with teflon-lined lid, protect from light, Cool, 4°C, Extract, 10 days of sample receipt, Analysis, 40 days after extraction	8 or 16 oz glass amber wide mouth with teflon-lined lid, Cool 4°C, Extract, 30 days Analysis, 45 days from date of collection
	Air	Not Applicable	Not Applicable	Not Applicable	PUF[6]: Store at 4°C, Extract, 14 days Analysis, 40 days after extraction
Petroleum Hydrocarbons	Water[10]	1 liter glass amber with teflon-lined lid, pH to ≤ 2 with H_2SO_4, Cool, 4°C, 28 days (oil & grease holding time),	Not Applicable	Not Applicable	DRO[7]: 1 liter glass with teflon-lined lid, pH to ≤ 2 with H_2SO_4, Cool, 4°C, Extract 7 days, Analysis 40 days GRO[8]: 40 ml glass VOA vial with teflon-lined septa, in triplicate without headspace. Cool, 4°C. pH ≤ 2 with HCl, 14 days
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable

Table 8.2-2
Organic Sample Containers, Preservatives, and Holding Times (continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2,3]	CLP 88 & 90[4, 5]	Other
Petroleum Hydrocarbons (continued)	Soil	Not Applicable	Not Applicable	Not Applicable	DRO[7]: 4 or 8 oz glass widemouth with teflon-lined lid, Cool, 4°C, Extract, 14 days Analysis, 40 days GRO[6]:4 oz widemouth glass jar with teflon-lined lid, with container filled as full as possible, Cool, 4°C, 14 days
	Air	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Purgeable Halocarbons	Water[10]	40 ml glass VOA vial (in duplicate) with teflon-lined septa with no headspace, Cool, 4°C, sodium thiosulfate if residual chlorine, 14 days	40 ml glass VOA vial (in duplicate) with teflon-lined septa with no headspace, Cool, 4°C, HCl or H_2SO_4 or solid $NaHSO_4$ to pH ≤ 2, sodium thiosulfate if residual chlorine, 14 days	Not Applicable	Not Applicable
	Liquid	Not Applicable	40 ml glass VOA vial (in duplicate) with teflon-lined septa, Cool, 4°C, 14 days	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	4 or 8 oz glass with teflon-lined lid, Cool, 4°C, TCLP Leachate: 14 days, Analysis: 14 days from end of TCLP leaching (not to exceed 28 days total)	Not Applicable	40 ml glass with teflon-lined septa, Cool 4°C, TCLP Leaching: 14 days Analysis: 14 days from end of TCLP leaching (not to exceed 28 days total)
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	4 or 8 oz glass container with teflon-lined lid, Cool 4°C, 14 days	Not Applicable	Not Applicable
	Soil	Not Applicable	4 or 8 oz glass container with teflon-lined lid, Cool 4°C, 14 days	Not Applicable	Not Applicable

Table 8.2-2
Organic Sample Containers, Preservatives, and Holding Times (continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2,3]	CLP 88 & 90[4,5]	Other
Semivolatiles	Water[12]	1 liter glass with teflon-lined lid, Cool, 4°C, Extract, 7 days Analysis, 40 days	1 liter amber glass with teflon-lined lid, Cool, 4°C, Extract, 7 days Analysis, 40 days	1 liter glass amber with teflon-lined lid, Cool, 4°C, Extract, 5 days from sample receipt Analysis, 40 days	1 liter glass amber with teflon-lined lid, Cool, 4°C, Extract, 7 days Analysis, 40 days
	Liquid	Not Applicable	100 ml glass with teflon lined lid, No preservative required, Extract, 14 days Analysis, 40 days	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	1 liter glass with teflon-lined lid, Cool, 4°C, TCLP Leaching: 14 days from field collection to TCLP extraction, Extract 7 days from TCLP extraction to preparative extraction, Analysis, 40 days from preparative extraction	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	8 or 16 oz glass wide mouth with teflon-lined lid, Cool, 4°C, Extract, 14 days Analysis, 40 days after extraction	Not Applicable	Not Applicable
	Soil	Not Applicable	8 or 16 oz glass widemouth with teflon-lined lid, Cool, 4°C, Extract, 14 days Analysis, 40 days after extraction	8 or 16 oz glass amber wide mouth with teflon-lined lid, Cool, 4°C, Extract, 10 days from sample receipt, Analysis, 40 days	Not Applicable
	Air	Not Applicable	Not Applicable	Not Applicable	PUF[m]/XAD: Store at 4°C, extract, 7 days, Analysis, 40 days

Table 8.2-2
Organic Sample Containers, Preservatives, and Holding Times
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2,3]	CLP 88 & 90[4, 5]	Other
Volatiles	Water[10]	40 ml glass VOA vial (in duplicate) with teflon-lined septa without headspace, Cool, 4°C, HCl to pH ≤ 2, 7 days with pH ≥ 2, Analyze within 14 days of collection with pH ≤ 2	40 ml glass VOA vial (in duplicate) with teflon-lined septa in duplicate without headspace, Cool, 4°C, HCl or H_2SO_4 or $NaHSO_4$ to pH ≤ 2, Sodium thiosulfate if residual chlorine, Analyze within 14 days of collection with pH ≤ 2	40 ml VOA glass vial (in duplicate) with teflon-lined septa, with no headspace, Cool, 4°C, 10 days from sample receipt	40 ml glass with teflon-lined septa, Cool, 4°C, HCl to pH 2, 14 days 40 ml glass with teflon-lined septa, Cool, 4°C, 7 days
	Liquid	Not Applicable	40 ml glass with teflon-lined septa, None required, 14 days	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	40 ml glass with teflon-lined septa, Cool to ≤ 4°C, TCLP Leaching: 14 days from field collection to TCLP extraction, Analysis: 14 days from preparative extraction to determinative analysis, (total lapsed time = 28 days)	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	4 or 8 oz glass with teflon-lined septa, Cool, 4°C, Analyze within 14 days	Applicable only to sediment: 4 or 8 oz glass with teflon-lined septa, Cool, 4°C, 10 days from sample receipt	Not Applicable
	Soil	Not Applicable	4 or 8 oz glass with teflon-lined septa, Cool, 4°C, Analyze within 14 days	4 or 8 oz glass with teflon-lined septa, Cool, 4°C, 10 days from sample receipt	Not Applicable

Table 8.2-2
Organic Sample Containers, Preservatives, and Holding Times (continued)

Analytical Parameters	Matrix	Methods			
		NPDES[1]	RCRA (SW846)[2,3]	CLP 88 & 90[4,5]	Other
Volatiles (Continued)	Air	Not Applicable	Not Applicable	Not Applicable	Summa Cannister: Store at ambient temperature, no holding time requirement VOST[10]: Store at < 0°C, 14 days [illegible][9]: Store at < 0°C, no holding time requirement

(1) National Pollutant Discharge Elimination System
(2) Resource Conservation and Recovery Act (Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd edition, Final Update I, July 1992
(3) Holding times are calculated from the date of collection
(4) Contract Laboratory Program
(5) Holding times are calculated from verified time of sample receipt
(6) Gasoline Range Organics
(7) Diesel Range Organics
(8) Polyurethane foam
(9) Industrial Hygiene
(10) Volatile Organic Sampling Train
(11) When a matrix spike (MS) analysis is required for a water sample, it should be sampled in duplicate (two separate sample containers). When a matrix spike/matrix spike duplicate (MS/MSD) analysis is required for a water sample, it should be sampled in triplicate (three separate sample containers).

TABLE 8.2-3
Radiological Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Recommended Containers[1]	Preservative	Maximum Holding Time	Minimum Volume Required for Analysis[2]
Gross Alpha/Beta	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	500 mls
	Soil	P, G	None		50[4] gms
Americium-241	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000[5] mls
	Soil	P, G	None		50[4] gms
Carbon-14	Water	P, G	Field adjusted to pH > 9 with NaOH[3]	180 days after collection	100 mls
	Soil	P, G	None		50[4] gms
Calcium-45	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	100 mls
Curium-242	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000[5] mls
	Soil	P, G	None		50[4] gms
Gamma Emmers Actinides, as applicable. Co-60, Cs-137, K-40, Mn-54, and other fission/activation products	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000[5] mls
	Soil	P, G	None		650[7] gms
Iron-55	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	50 mls
Lead-210	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	500 mls
	Soil	P, G	None		50[4] gms
Neptunium-237	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000[5] mls
	Soil	P, G	None		50[4] gms

TABLE 8.2-3
Radiological Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Recommended Containers[1]	Preservative	Maximum Holding Time	Minimum Volume Required for Analysis[2]
Promethium-147	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	250 mls
Plutonium-238,239/240	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000^5 mls
	Soil	P, G	None		50^4 gms
Radium-226	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000^5 mls
	Soil	P, G	None		50^4 gms
Radium-228	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000^5 mls
	Soil	P, G	None		50^4 gms
Strontium-89,90 and Total Strontium	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000^5 mls
	Soil	P, G	None		50^4 gms
Technetium-99	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	100 mls
	Soil	P, G	None		50 gms
Thorium-227,228,230,232	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000^5 mls
	Soil	P, G	None		50^4 gms
Total Uranium	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	50 mls
	Soil	P, G	None		50^4 gms
Tritium	Water	P, G[6]	None	180 days after collection	100 mls
	Soil	P, G[6]	None		100 gms

TABLE 8.2-3
Radiological Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix	Recommended Containers[1]	Preservative	Maximum Holding Time	Minimum Volume Required for Analysis[2]
Uranium-233/234,235/236	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000[3] mls
	Soil	P, G	None		50[4] gms
Uranium-238	Soil	P, G	None	180 days after collection	50[4] gms

1 Plastic (polyethylene), Glass

2 Assumes that quality control samples have been assigned in the field. If duplicates, matrix spikes and/or matrix spike duplicates are to be assigned by the laboratory additional multiple sample volumes are required. Volumes listed are for standard aliquot size. Detection limit requirements may necessitate larger volumes.

3 Assumes that Carbon is in the form of CO_3^-.

4 May be aliquoted or sequentially determined from the same volume.

5 May be aliquoted or sequentially determined from the same volume.

6 Tritium is very volatile. Sample containers must by air tight to eliminate tritium loss.

7 Dry weight.

TABLE 8.4-1
Summary of Inorganic Method Calibrations

Analysis	Calibration	Methods			
		NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
Acidity	Initial	2 point calibration of pH meter (± 0.05 pH units of true value)	Not Applicable	Not Applicable	2 point calibration of pH meter (± 0.05 pH units of true value)
	Continuing	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Ending	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Alkalinity	Initial	2 point calibration of pH meter (± 0.05 pH units of true value)	Not Applicable	Not Applicable	2 point calibration of pH meter (± 0.05 pH units of true value)
	Continuing	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Ending	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Ammonia	Initial	7 levels plus blank. "r"[4] ≥ 0.995	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 level every 10 samples ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Ending	1 level ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Ammonia (TKN)	Initial	7 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 level every 10 samples ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Ending	1 level ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
BOD	Initial	Winkler calibration	Not Applicable	Not Applicable	Winkler calibration
	Continuing	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Ending	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable

Table 8.4-1
Summary of Inorganic Method Calibrations
(continued)

Analysis	Methods				
	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
Bromide (Titration and Spectro-photometric)	Initial	2 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable	8 levels plus blank "r"[4] ≥ 0.995
	Continuing	Single point daily and after every 10 samples ± 15% of true value	Not Applicable	Not Applicable	1 point every 10 samples ± 15% of true value
	Ending	1 level ± 15% of true value	Not Applicable	Not Applicable	1 point every 10 samples ± 15% of true value
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Bromide (IC)	Initial	3 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable	Not Applicable
	Continuing	Single point daily and after every 20 samples ± 10% of true value	Not Applicable	Not Applicable	Not Applicable
	Ending	1 level ± 10% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cation Exchange Capacity	See ICAP Calibration Procedures, this is an extraction method.				
Chemical Oxygen Demand (COD)	Initial	3 levels plus blank	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 point every 10 samples ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Ending	1 point ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Chloride (IC)	Initial	3 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable	Not Applicable
	Continuing	Single point daily and after every 20 samples ± 10% of true value	Not Applicable	Not Applicable	Not Applicable

Table 8.4-1
Summary of Inorganic Method Calibrations
(continued)

Analysis	Calibration	Methods			
		NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
Chloride (IC) (continued)	Ending	1 level ± 10% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Chloride (LACHAT)	Initial	8 levels plus blank "r"[4] ≥ 0.995	3 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable
	Continuing	1 point every 10 samples ± 15% of true value	1 standard every 15 samples ± 15% of true value	Not Applicable	Not Applicable
	Ending	1 level ± 15% of true value	1 level ± 15% of true value	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Chlorine, Residual	Initial	Standardize titrant	Not Applicable	Not Applicable	Not Applicable
	Continuing		Not Applicable	Not Applicable	Not Applicable
	Ending		Not Applicable	Not Applicable	Not Applicable
	Other		Not Applicable	Not Applicable	Not Applicable
Chromium (Hexavalent) Cr^{+6}	Initial	4 standards plus blank "r"[4] ≥ 0.995	Standard concentration ranging from 0.5 to 5 mg/L Cr(VI)	Not Applicable	7 standards plus blank "r"[4] ≥ 0.995
	Continuing	1 point every 10 samples ± 15% of true value	1 independently-prepared check standard every 15 samples	Not Applicable	1 point every 10 samples ± 15% of true value
	Ending	1 point ± 15% of true value	Not Applicable	Not Applicable	1 point ± 15% of true value
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Color	Initial	3 standards plus blank	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 point every 10 samples	Not Applicable	Not Applicable	Not Applicable
	Ending	1 point	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Conductivity	Initial	Standardize meter with 0.01 M KCl	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 point every 10 samples	Not Applicable	Not Applicable	Not Applicable

Table 8.4-1
Summary of Inorganic Method Calibrations
(continued)

Analysis	Calibration	Methods			
		NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
Conductivity (continued)	Ending	1 point ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	1 point ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
Cyanide (Amenable)	Initial	3 levels plus blank r^{2}[4] ≥ 0.995	6 levels plus blank r^{2}[4] ≥ 0.995	Not Applicable	8 levels plus blank r^{2}[4] ≥ 0.995
	Continuing	1 level every 10 samples ± 15% of true value	1 mid-level every 10 samples ± 15 % of true value	Not Applicable	1 level every 10 samples ± 15% of true value
	Ending	1 level ± 15 % of true value	1 level ± 15 % of true value	Not Applicable	1 level ± 15 % of true value
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cyanide (Free)	Initial	Not Applicable	Not Applicable	Not Applicable	8 levels plus blank r^{2}[4] ≥ 0.995
	Continuing	Not Applicable	Not Applicable	Not Applicable	1 level every 10 samples ± 15 % of true value
	Ending	Not Applicable	Not Applicable	Not Applicable	1 level ± 15 % of true value
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cyanide (Total)	Initial	6 levels plus blank r^{2}[4] ≥ 0.995	6 levels plus blank r^{2}[4] ≥ 0.995	minimum of 8 levels plus blank r^{2}[4] ≥ 0.995	8 levels plus blank r^{2}[4] ≥ 0.995
	Continuing	1 mid-level every 10 samples ± 15 % of true value	1 level every 10 samples ± 15 % of true value	1 level every 10 samples ± 15 % of true value	1 mid-level every 10 samples ± 15 % of true value
	Ending	1 level ± 15 % of true value	1 level ± 15 % of true value	1 level ± 15 % of true value	1 level ± 15 % of true value
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable

ADDITION TO:
TABLE 8.4-1
Summary of Inorganic Method Calibrations

Analysis	Methods				
	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
Hydrazine Colorimetric	Initial	Not Applicable	Not Applicable	Not Applicable	6 levels plus blank "r"[4] ≥ 0.995
	Continuing	Not Applicable	Not Applicable	Not Applicable	1 level every 10 samples ± 15 % of true value
	Ending	Not Applicable	Not Applicable	Not Applicable	1 level ± 15 % of true value
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable

Table 8.4-1
Summary of Inorganic Method Calibrations
(continued)

Analysis	Methods				
	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
Flashpoint (Ignitability)	Initial	Not Applicable	p-Xylene reference standard must have flashpoint of 27.2°C ± 1.1°C	Not Applicable	Not Applicable
	Continuing	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Ending	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Fluoride (IC)	Initial	3 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 level every 20 samples ± 10% of true value	Not Applicable	Not Applicable	Not Applicable
	Ending	1 level ± 10% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Fluoride (Ion-Specific Electrode)	Initial	5-8 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 mid-level every 10 samples ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Ending	1 level ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Hardness	Initial	Standardize titrant	Not Applicable	Not Applicable	Hardness by calculation using ICP results
	Continuing	Not Applicable	Not Applicable	Not Applicable	
	Ending	Not Applicable	Not Applicable	Not Applicable	
	Other	Not Applicable	Not Applicable	Not Applicable	
Iodide (Titration)	Initial	2 points plus blank ± 0.5 N	Not Applicable	Not Applicable	2 points plus blank ± 0.5 N
	Continuing	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Ending	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable

Table 8.4-1
Summary of Inorganic Method Calibrations
(continued)

Analysis	Methods				
	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
Methylene Blue Active Substances (MBAS)	Inital	9 points plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable	6 points plus blank "r"[4] ≥ 0.995
	Continuing	Not Applicable	Not Applicable	Not Applicable	1 level every 10 samples ± 15% of true value
	Ending	Not Applicable	Not Applicable	Not Applicable	1 level ± 15% of true value
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Nitrogen, Nitrate-Nitrite	Initial	8 levels plus blank "r"[4] ≥ 0.995	Nitrate: 6 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable
	Continuing	1 level every 10 samples ± 15% of true value	1 level every 10 samples ± 15% of true value	Not Applicable	Not Applicable
	Ending	1 level ± 15% of true value	1 level ± 15% of true value	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Odor	Initial	No calibration	Not Applicable	Not Applicable	Not Applicable
	Continuing	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Ending	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Oil and Grease	Initial	This is a gravimetric determination. Calibrate balance before use.	This is a gravimetric determination. Calibrate balance before use.	Not Applicable	3 points plus blank "r"[4] ≥ 0.995
	Continuing			Not Applicable	1 point ± 15% of true value
	Ending			Not Applicable	1 point ± 15% of true value
	Other			Not Applicable	Not Applicable
pH	Initial	2-3 point calibration (± 0.05 pH units of true value)	2 point calibration (± 0.05 pH units of true value)	2 point calibration (± 0.05 pH units of true value)	Not Applicable
	Continuing	Not Applicable	Not Applicable	Not Applicable	Not Applicable

Table 8.4-1
Summary of Inorganic Method Calibrations
(continued)

Analysis	Methods				
	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
pH (continued)	Other	Third point check	Third point check	Third point check	Not Applicable
Phenolics (Automated)	Initial	3 levels plus blank "r"[4] ≥ 0.995	3 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable
	Continuing	1 level every 10 samples ± 15% of true value	1 level every 10 samples ± 15% of true value	Not Applicable	Not Applicable
	Ending	1 level ± 15% of true value	1 level ± 15% of true value	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Phenolics (Manual)	Initial	5 levels plus blank "r"[4] ≥ 0.995	5 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable
	Continuing	1 level every 10 samples ± 15% of true value	1 level every 10 samples ± 15% of true value	Not Applicable	Not Applicable
	Ending	1 level ± 15% of true value	1 level ± 15% of true value	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Phosphorous (Total and Ortho-phosphate) (LACHAT)	Initial	7 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 level every 10 samples ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Ending	1 level ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Phosphorous (Ortho-phosphate) by IC	Initial	3 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable	Not Applicable
	Continuing	Single point daily and after every 20 samples ± 10% of true value	Not Applicable	Not Applicable	Not Applicable

Table 8.4-1
Summary of Inorganic Method Calibrations
(continued)

Analysis	Calibration	Methods			
		NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
Phosphorous (Ortho-phosphate) by IC (continued)	Ending	1 level ± 10% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Particulate/ Air	Initial	Not Applicable	Not Applicable	Not Applicable	Initial balance check with Class S weights
	Continuing	Not Applicable	Not Applicable	Not Applicable	Check balance with Class S weights every 15 filters
	Ending	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Silica	Initial	6 standards plus blank $r^{2(4)} \geq 0.995$	Not Applicable	Not Applicable	7 standards plus blank $r^{2(4)} \geq 0.995$
	Continuing	1 level ± 15% of true value	Not Applicable	Not Applicable	1 level ± 15% of true value
	Ending	1 level ± 15% of true value	Not Applicable	Not Applicable	1 level ± 15% of true value
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Sulfate (IC)	Initial	3 levels plus blank $r^{2(4)} \geq 0.995$	3 levels plus blank $r^{2(4)} \geq 0.995$	Not Applicable	Not Applicable
	Continuing	1 level every 20 samples ± 10% of true value	1 level every 15 samples ± 15% of true value	Not Applicable	Not Applicable
	Ending	1 level ± 10% of true value	1 level ± 15% of true value	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Sulfate (LACHAT)	Initial	7 levels plus blank $r^{2(4)} \geq 0.995$	Not Applicable	Not Applicable	Not Applicable
	Continuing	Single point daily and after every 10 samples ± 15% of true value	Not Applicable	Not Applicable	Not Applicable

Table 8.4-1
Summary of Inorganic Method Calibrations
(continued)

Analysis	Methods				
	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
pH (continued)	Other	Third point check	Third point check	Third point check	Not Applicable
Phenolics (Automated)	Initial	3 levels plus blank "r"[4] ≥ 0.995	3 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable
	Continuing	1 level every 10 samples ± 15% of true value	1 level every 10 samples ± 15% of true value	Not Applicable	Not Applicable
	Ending	1 level ± 15% of true value	1 level ± 15% of true value	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Phenolics (Manual)	Initial	5 levels plus blank "r"[4] ≥ 0.995	5 levels plus blank "r"[4] ≥ 0.995		
	Continuing	1 level every 10 samples ± 15% of true value	1 level every 10 samples ± 15% of true value		
	Ending	1 level ± 15% of true value	1 level ± 15% of true value		
	Other	Not Applicable	Not Applicable		
Phosphorous (Total and Ortho-phosphate) (LACHAT)	Initial	7 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 level every 10 samples ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Ending	1 level ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Phosphorous (Ortho-phosphate) by IC	Initial	3 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable	Not Applicable
	Continuing	Single point daily and after every 20 samples ± 10% of true value	Not Applicable	Not Applicable	Not Applicable

Table 8.4-1
Summary of Inorganic Method Calibrations
(continued)

Analysis	Calibration	Methods			
		NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
Phosphorous (Ortho-phosphate) by IC (continued)	Ending	1 level ± 10% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Particulate/ Air	Initial	Not Applicable	Not Applicable	Not Applicable	Initial balance check with Class S weights
	Continuing	Not Applicable	Not Applicable	Not Applicable	Check balance with Class S weights every 15 filters
	Ending	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Silica	Initial	6 standards plus blank $r^{*[4]} \geq 0.995$	Not Applicable	Not Applicable	7 standards plus blank $r^{*[4]} \geq 0.995$
	Continuing	1 level ± 15% of true value	Not Applicable	Not Applicable	1 level ± 15% of true value
	Ending	1 level ± 15% of true value	Not Applicable	Not Applicable	1 level ± 15% of true value
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Sulfate (IC)	Initial	3 levels plus blank $r^{*[4]} \geq 0.995$	3 levels plus blank $r^{*[4]} \geq 0.995$	Not Applicable	Not Applicable
	Continuing	1 level every 20 samples ± 10% of true value	1 level every 15 samples ± 15% of true value	Not Applicable	Not Applicable
	Ending	1 level ± 10% of true value	1 level ± 15% of true value	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Sulfate (LACHAT)	Initial	7 levels plus blank $r^{*[4]} \geq 0.995$	Not Applicable	Not Applicable	Not Applicable
	Continuing	Single point daily and after every 10 samples	Not Applicable	Not Applicable	Not Applicable

INTERNATIONAL TECHNOLOGY CORPORATION

Procedure Change No.:

OSQAMP-02

IT ANALYTICAL SERVICES DIVISION
PROCEDURE/DOCUMENT CHANGE

PROCEDURE/DOCUMENT NUMBER: N/A			
PROCEDURE/DOCUMENT TITLE: ITAS Operation-Specific Quality Assurance Management Plan (OS QAMP) (Revision 0, September 1, 1993)			
PROCEDURE/DOCUMENT SECTION(S) AFFECTED BY CHANGE: Table 8.4-1, "Summary of Inorganic Method Calibrations", page 165.			
REASON FOR ADDITION OR CHANGE: Typographical error in table.			
CHANGE EFFECTIVE FROM:	12/14/93	TO:	Ongoing
SAMPLES OR PROJECTS AFFECTED: N/A			
CHANGE: Replace page 165 with the attached corrected page. The ending calibration acceptance criteria for sulfide is changed from ±10 percent to ±15 percent.			
SUBMITTED BY/DATE: Patti Carswell 12/14/93			
APPROVED BY:			

Patti Carswell 12/14/93	TECHNICAL SPECIALIST/DATE
Brian E Lewis 12/14/93	ITAS DIRECTOR, HEALTH & SAFETY/DATE
Paul C Hall 12/14/93	ITAS DIRECTOR, QA/QC/DATE

Table 8.4-1
Summary of Inorganic Method Calibrations
(continued)

Analysis	Methods				
	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
Sulfate (LACHAT) (continued)	Ending	1 level ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Sulfate (Turbidimetric)	Initial	6 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 point ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Ending	1 point ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Sulfide (Titration)	Initial	2 levels plus blank ± 0.5 N	This is a colorimetric titration. Therefore, calibrations are not applicable.	Not Applicable	Not Applicable
	Continuing	1 point every 10 samples ± 15% of true value		Not Applicable	Not Applicable
	Ending	1 level ± 15% of true value		Not Applicable	Not Applicable
	Other	Not Applicable		Not Applicable	Not Applicable
Sulfide	Initial	5 levels "r"[4] ≥ 0.995	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 point every 10 samples ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Ending	1 level ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Sulfite	Initial	This is a colorimetric titration. Therefore, calibrations are not applicable.	Not Applicable	Not Applicable	Not Applicable
	Continuing		Not Applicable	Not Applicable	Not Applicable
	Ending		Not Applicable	Not Applicable	Not Applicable
	Other		Not Applicable	Not Applicable	Not Applicable

Table 8.4-1
Summary of Inorganic Method Calibrations
(continued)

Analysis	Methods				
	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
Total Organic Carbon (TOC)	Initial	3 levels plus blank "r"[4] ≥ 0.995	3 levels plus blank	Not Applicable	5 levels plus blank
	Continuing	LCS every 20 samples ± 15% of true value	LCS every 20 samples ± 15% of true value	Not Applicable	LCS every 20 samples ± 15% of true value
	Ending	1 level ± 15% of true value	1 level ± 15% of true value	Not Applicable	1 level ± 15% of true value
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Total Organic Halides (TOX)	Initial	Analyze instrument calibration standard in duplicate plus a blank	Analyze instrument calibration standard in duplicate plus a blank	Not Applicable	Not Applicable
	Continuing	Reanalysis of instrument-calibration standard after each group of 8 pyrolysis determinations	Reanalysis of instrument-calibration standard after each group of 8 pyrolysis determinations	Not Applicable	Not Applicable
	Ending	Not Applicable	1 level ± 15% of true value	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Total Solids (Dissolved and Volatile)	Initial	This is a gravimetric determination. Calibrate balance before use.	Not Applicable	Not Applicable	Not Applicable
	Continuing		Not Applicable	Not Applicable	Not Applicable
	Ending		Not Applicable	Not Applicable	Not Applicable
	Other		Not Applicable	Not Applicable	Not Applicable
Total Suspended Solids	Initial	This is a gravimetric determination. Calibrate balance before use.	Not Applicable	Not Applicable	Not Applicable
	Continuing		Not Applicable	Not Applicable	Not Applicable
	Ending		Not Applicable	Not Applicable	Not Applicable
	Other		Not Applicable	Not Applicable	Not Applicable
Turbidity	Initial	Minimum of 1 level in each instrument range Follow manufacturer's instructions	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 level	Not Applicable	Not Applicable	Not Applicable
	Ending	1 level	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable

Table 8.4-1
Summary of Inorganic Method Calibrations
(continued)

Analysis	Calibration	Methods			
		NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
Sulfate (LACHAT) (continued)	Ending	1 level ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Sulfate (Turbidimetric)	Initial	6 levels plus blank "r"[4] ≥ 0.995	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 point ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Ending	1 point ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Sulfide (Titration)	Initial	2 levels plus blank ± 0.5 N	This is a colorimetric titration. Therefore, calibrations are not applicable.	Not Applicable	Not Applicable
	Continuing	1 point every 10 samples ± 15% of true value		Not Applicable	Not Applicable
	Ending	1 level ± 15% of true value		Not Applicable	Not Applicable
	Other	Not Applicable		Not Applicable	Not Applicable
Sulfide	Initial	5 levels "r"[4] ≥ 0.995	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 point every 10 samples ± 15% of true value	Not Applicable	Not Applicable	Not Applicable
	Ending	1 level ± 10% of true value	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Sulfite	Initial	This is a colorimetric titration. Therefore, calibrations are not applicable.	Not Applicable	Not Applicable	Not Applicable
	Continuing		Not Applicable	Not Applicable	Not Applicable
	Ending		Not Applicable	Not Applicable	Not Applicable
	Other		Not Applicable	Not Applicable	Not Applicable

Table 8.4-1
Summary of Inorganic Method Calibrations
(continued)

Analysis	Calibration	Methods			
		NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
Total Organic Carbon (TOC)	Initial	3 levels plus blank $r^{2(4)} \geq 0.995$	3 levels plus blank	Not Applicable	5 levels plus blank
	Continuing	LCS every 20 samples ± 15% of true value	LCS every 20 samples ± 15% of true value	Not Applicable	LCS every 20 samples ± 15% of true value
	Ending	1 level ± 15% of true value	1 level ± 15% of true value	Not Applicable	1 level ± 15% of true value
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Total Organic Halides (TOX)	Initial	Analyze instrument calibration standard in duplicate plus a blank	Analyze instrument calibration standard in duplicate plus a blank	Not Applicable	Not Applicable
	Continuing	Reanalysis of instrument-calibration standard after each group of 8 pyrolysis determinations	Reanalysis of instrument-calibration standard after each group of 8 pyrolysis determinations	Not Applicable	Not Applicable
	Ending	Not Applicable	1 level ± 15% of true value	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Total Solids (Dissolved and Volatile)	Initial	This is a gravimetric determination. Calibrate balance before use.	Not Applicable	Not Applicable	Not Applicable
	Continuing		Not Applicable	Not Applicable	Not Applicable
	Ending		Not Applicable	Not Applicable	Not Applicable
	Other		Not Applicable	Not Applicable	Not Applicable
Total Suspended Solids	Initial	This is a gravimetric determination. Calibrate balance before use.	Not Applicable	Not Applicable	Not Applicable
	Continuing		Not Applicable	Not Applicable	Not Applicable
	Ending		Not Applicable	Not Applicable	Not Applicable
	Other		Not Applicable	Not Applicable	Not Applicable
Turbidity	Initial	Minimum of 1 level in each instrument range Follow manufacturer's instructions	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 level	Not Applicable	Not Applicable	Not Applicable
	Ending	1 level	Not Applicable	Not Applicable	Not Applicable
	Other				

Table 8.4-1
Summary of Inorganic Method Calibrations
(continued)

Analysis	Methods				
	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
GFAA and Flame AA Metals	Initial	3 levels plus blank "r"[4] ≥ 0.995	3 levels plus blank "r"[4] ≥ 0.995	3 levels plus blank (one standard at CRDL) "r"[4] ≥ 0.995	3 levels plus blank "r"[4] ≥ 0.995
	Continuing	1 level every 10 samples ± 10% of true value	1 level every 20 samples ± 20% of true value	Every 10 samples or every 2 hours, which ever is more frequent 1 level ± 10% of true value	1 level every 10 samples ± 10% of true value
	Ending	1 level ± 10% of true value	1 level ± 20% of true value	1 level ± 10% of true value	1 level ± 10% of true value
	Other	Quarterly - Instrument detection limits	Quarterly - Instrument detection limits	Daily - CRDL standards at the beginning of each run Quarterly - Instrument detection limits	Quarterly - Instrument detection limits
Mercury by CVAA	Initial	5 levels plus blank "r"[4] ≥ 0.995	5 levels plus blank "r"[4] ≥ 0.995	4 levels plus blank "r"[4] ≥ 0.995	5 levels plus blank "r"[4] ≥ 0.995
	Continuing	1 level daily or every 10 samples, whichever is more frequent ± 10% of true value	1 level every 10 samples ± 10% of original prepared standard	1 level every 10 samples or every 2 hours, which ever is more frequent ± 20 % of true value	1 level every 10 samples ± 10% of original prepared standard
	Ending	1 level ± 10% of true value	1 level ± 10% of original prepared standard	1 level ± 20 % of true value	1 level ± 10% of original prepared standard
	Other	Quarterly - Instrument detection limits	Quarterly - Instrument detection limits	Daily - CRDL standards at the beginning of each sequence Quarterly - Instrument detection limits	Quarterly - Instrument detection limits
ICP Metals	Initial	1 level and blank	1 level and blank	1 level and blank	1 level and blank
	Continuing	1 level every 10 samples ± 5% of true value	1 level every 10 samples ± 10% of true value	1 level every 10 samples or every 2 hours, whichever is more frequent	1 level every 10 samples ± 10% of true value

Table 8.4-1
Summary of Inorganic Method Calibrations
(continued)

Analysis	Methods				
	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP (88 & 90)[3]	OTHER
ICP Metals (continued)	Ending	1 level ± 5% of true value	± 10% of true value	± 10% of true value	± 10% of true value
	Other	Quarterly - Instrument detection limits Annually - ICP interelement correction factors ICSA, ICSAB: Analyze at beginning and end of every 8 hours	Quarterly - Instrument detection limits Annually - ICP interelement correction factors ICSA, ICSAB: Analyze at beginning and end of every 8 hours	Daily - CRI standards at the beginning and end of each sequence or every 8 hours, whichever is more frequent Quarterly - Instrument detection limits Annually - ICP interelement correction factors Quarterly - Linear Ranges ICSA, ICSAB: Analyze at beginning and end of every 8 hours	Quarterly - Instrument detection limits Annually - ICP interelement correction factors ICSA, ICSAB: Analyze at beginning and every 8 hours

(1) National Pollutant Discharge Elimination System
(2) Resource Conservation and Recovery Act (Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd edition, Final Update I, July 1992
(3) Contract Laboratory Program
(4) "r" = correlation coefficient

TABLE 8.4-2
Summary of Organic Method Calibrations

Analysis	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP 88[3]	CLP 90[3]	OTHER
Aromatic Volatiles by GC	Initial	Minimum of 3 levels, lowest near but above MDL. If % RSD[4] < 10, assume linearity and average RF[5] used. Otherwise, plot calibration curve.	Minimum of 5 levels, lowest near but above MDL. If % RSD[4] < 20, linearity is assumed and average RF[5] is used. Otherwise, plot calibration curve.	Not Applicable	Not Applicable	5 levels If RSD[4] ≤ 20%, assume linearity. If > 20%, plot calibration curve.
	Continuing	1 or more calibration standards analyzed daily. Compare results to method calibration table.	Mid-level calibration standard analyzed every 10 samples. If not within ± 15 % of predicted response, recalibrate.	Not Applicable	Not Applicable	1 every 10 samples, % D[5] ≤ 15% 1 every 10 samples, % D ≤ 10% (≤ 15% for surrogates)
	Ending	Not Applicable	Retention times in windows for all compounds	Not Applicable	Not Applicable	Not Applicable
	Other	When doubt exists in compound identification, second column confirmation is recommended.	When doubt exists in compound identification, second column confirmation is recommended. Retention time windows established over 72-hour period by injection of each compound three times.	Not Applicable	Not Applicable	Not Applicable
Dioxins/ Dibenzofurans by HRGC/ LRMS[7]	Initial	3 levels If %RSD[4] <10%, use mean RF[5]. If %RSD ≥ 10%, plot calibration curve point to point	5 levels in triplicate % RSD[4] ≤ 15%	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 level each working day. %D[5] must be ≤10%. If not, analyze fresh standard. If still not, recalibrate	1 level every 12 hours after window performance mix. Standard must have RFs[5] with %D[5] ≤ 30% from initial	Not Applicable	Not Applicable	Not Applicable
	Ending	Not Applicable	Window	Not Applicable	Not Applicable	Not Applicable

Table 8.4-2
Summary of Organic Method Calibrations
(continued)

Analysis	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP 88[3]	CLP 90[3]	OTHER
Dioxins/ Dibenzofurans by HRGC/ LRMS[7] (continued)	Other	Not Applicable	Window mix to set congener windows every 12 hours at beginning of sequence. Isotope ratios in standard must meet criteria in method. Valley between 2,3,7,8-TCDD[6] and 1,2,3,4-TCDD must be ≤ 25% of the 2,3,7,8-TCDD peak height.	Not Applicable	Not Applicable	Not Applicable
Dioxins/ Dibenzofurans by HRGC/ HRMS[8]	Initial	Not Applicable	5 levels plus window defining solution. %RSD[4] for natives ≤20% for RFs[9]; %RSD, for labelled compounds ≤30% for RFs.	Not Applicable	Not Applicable	Not Applicable
	Continuing	Not Applicable	1 level every 12 hours after window defining solution. RFs[9] with %D[5] ≤20% for natives; %D ≤30% for labelled compounds from initial	Not Applicable	Not Applicable	Not Applicable
	Ending	Not Applicable	1 level: RFs[9] with %D ≤20% for natives; %D[5] ≤30% for labelled compounds from initial	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Isotope ratios in standard must meed criteria in method. Valley between 2,3,7,8-TCDD[6] and all other TCDDs must be ≤25% of the 2,3,7,8-TCDD height	Not Applicable	Not Applicable	Not Applicable
Formaldehyde	Initial	Not Applicable	Not Applicable	Not Applicable	Not Applicable	5 point calibration RSD[4] ≤ 20% Recalibrate if

Table 8.4-2
Summary of Organic Method Calibrations
(continued)

Analysis	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP 88[3]	CLP 90[3]	OTHER
Formaldehyde (continued)	Continuing	Not Applicable	Not Applicable	Not Applicable	Not Applicable	1 per 10 samples %D[5] ≤ 15% Reanalyze and recalibrate if criteria not met
	Ending	Not Applicable	Not Applicable	Not Applicable	Not Applicable	At end of run %D[5] ≤ 15% Reanalyze and recalibrate if criteria not met
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Herbicides by GC	Initial	Minimum of 3 levels, lowest near but above MDL. If % RSD[4] ≤ 10 %, use avg. RF[9]. Otherwise, plot calibration curve	Minimum of 5 levels, lowest near but above MDL. If % RSD[4] ≤ 20%, use avg. RF[9]. Otherwise, plot calibration curve	Not Applicable	Not Applicable	Minimum 3 levels, %RSD[4] ≤20% (use mean RF[9]) 1 level calibration standard at beginning and end of each day (must be different levels) %D[5] must be ± 20% of initial or recalibrate
	Continuing	1 or more calibration standards analyzed daily. If not within ± 10% of predicted response, recalibrate	Mid-level calibration standard run every 10 samples. If not within ± 15 % of predicted response, recalibrate Retention times in windows for all continuing standards	Not Applicable	Not Applicable	%D[5] must be ≤20% or recalibrate 2 levels every 24 hours. One at beginning and one at the end
	Ending	Not Applicable	Retention times in windows for all compounds.	Not Applicable	Not Applicable	%D[5] ≤ 20%

Table 8.4-2
Summary of Organic Method Calibrations
(continued)

Analysis	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP 88[3]	CLP 90[3]	OTHER
Herbicides by GC (continued)	Other	When doubt exists in compound identification, second column confirmation should be used	When doubt exists in compound identification, second column confirmation should be used Retention time windows established over 72 hour period by injection of each compound three times	Not Applicable	Not Applicable	Not Applicable
Nitroaromatics by HPLC	Initial	Not Applicable	Analyze standards in triplicate. Curve should be linear with zero intercept.	Not Applicable	Not Applicable	Minimum of 5 levels ≤ 20% RSD[4] avg to be used. Otherwise point to point calculations if curve ≤ 30% RSD
	Continuing	Not Applicable	Midpoint calibration standard after the midpoint of sample run. Must agree within 20% of initial values, otherwise reinject all solutions in triplicate and recalculate calibration curve.	Not Applicable	Not Applicable	Single point for each analyte at the beginning of each sequence or each 24 hours. ≤ 15% D[5] from initial for each analyte. Every single component after every 10 or less sample injections ≤ 15% D[5] from initial for each analyte. Retention times in windows for in all continuing standards

Table 8.4-2
Summary of Organic Method Calibrations
(continued)

Analysis	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP 88[3]	CLP 90[3]	OTHER
Nitroaromatics by HPLC (continued)	Ending	Not Applicable	Midpoint calibration standard after the end of sample run. Must agree within 20% of initial values, otherwise reinject all solutions in triplicate and recalculate calibration curve.	Not Applicable	Not Applicable	≤ 15% D[5] from initial for each analyte.
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Retention time windows established over 72 hour period by injection of each compound three times
Non-Methane Organic Compounds (NMOC)	Initial	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Minimum of 3 levels %RSD[4] must be ≤ 25 %
	Continuing	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Mid-level standard every 24 hours % D[5] must be ≤ 20 % from initial or recalibration required
	Ending	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PAHs by HPLC	Initial	Minimum of 3 levels, lowest near but above MDL. If %RSD[4] < 10, assume linearity and average RF is used. Otherwise, plot calibration curve.	Minimum of 5 levels, lowest near but above MDL. If %RSD[4] < 20, assume linearity and average RF is used. Otherwise, plot calibration curve.	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 or more calibration standards analyzed daily. If not within ± 15% of predicted response, recalibrate.	Mid-level calibration standard analyzed every 10 samples. If not within ± 15% of predicted response, recalibrate.	Not Applicable	Not Applicable	Not Applicable

Table 8.4-2
Summary of Organic Method Calibrations
(continued)

Analysis	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP 88[3]	CLP 90[3]	OTHER
PAHs by HPLC (continued)	Ending	Not Applicable	Retention times in windows for all compounds.	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Retention time windows established over 72-hour period by injection of each compound three times.	Not Applicable	Not Applicable	Not Applicable
PCBs by HRMS	Initial	Not Applicable	Not Applicable	Not Applicable	Not Applicable	5 Levels %RSD ≤ 30% for RFs
	Continuing	Not Applicable	Not Applicable	Not Applicable	Not Applicable	1 Level every 12 hours. RFs[5] ≤ 30% for natives after window defining solution
	Ending	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Pesticides/ PCBs	Initial	Minimum of 3 levels, lowest near but above MDL. If % RSD[4] ≤ 10% use avg. RF[5]. Otherwise plot calibration curve	Minimum of 5 levels, lowest near but above MDL. If % RSD[4] ≤ 20 use average RF[5]. Otherwise plot calibration curve. Check for DDT[11] and endrin degradation. % breakdown must be < 20.	3 levels each time a new column is used. Beginning of 72-hour sequence: Evaluation Mixes: Linearity ≤ 10% Degradation ≤ 20%. Individual Standard Mixes to establish RTs[21] and CFs[12]	3 levels (except multi-components at one level), the lowest at the CRQL[16]. The RSD[4] must be ≤ 20 %, however, 2 compounds may be > 20 % but must be < 30 %. Analyze Resolution Check Mixture and PEM[13] prior to initial calibration.	Method 505: Minimum of 3 standards: % RSD[4] ≤ 20% Method 508A: Minimum of 5 standards: The RSD[4] of RF[5] must be < 6% for the 7 injections at 0.1 ng/uL level

Table 8.4-2
Summary of Organic Method Calibrations
(continued)

Analysis	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP 88[3]	CLP 90[3]	OTHER
Pesticides/ PCBs (continued)	Continuing	1 or more calibration standards analyzed daily. If not within ± 10 % of predicted response, recalibrate.	Mid-level calibration standard analyzed every 10 samples; must agree within ± 15 % of predicted response or recalibrate	Evaluation Mix B and Individual Standard Mixes (A or B) run alternately between every 5 samples Individual Standard Mixes: CFs ≤ 15% D[5] for quantitation, ≤ 20% D[5] for confirmation, retention times in windows Evaluation Mix: Degradation ≤ 20 %	Instrument Blank and midpoint calibration or PEM[13] standard are run once per 12 hour period. Standard must be within ± 25 % of predicted response. Check for DDT[11] and endrin degradation every 12 hours. % breakdown must be ≤ 20.	Method 505: 1 or more levels every 24 hours. %D[5] ± 20% or less: if not, recalibrate with fresh standards Method 508A: 1 standard at 0.1 ng/uL must be analyzed at least once after each 8 hours of operation. %D[5] must be ± 20%
	Ending	Not Applicable	Retention times in windows for all compounds	72-hour sequence must end with Individual Standards A and B.	Samples must be bracketed by acceptable Instrument Blank, PEM[13], and both Individual Standard Mixtures A and B.	Not Applicable
	Other	When doubt exists in compound identification, second column confirmation should be used.	When doubt exists in compound identification, second column confirmation should be used. Retention time windows established over 72 hour period by injection of each compound three times	Retention time windows established over 72 hour period by injection of each compound three times	Second column confirmation is mandatory.	Not Applicable
Petroleum Hydrocarbons, Total Recoverable, by IR	Initial	Minimum of 3 levels plus blank "r"[14] ≥ 0.995	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Continuing	1 level Expected response should be within ± 15%	Not Applicable	Not Applicable	Not Applicable	Not Applicable

Table 8.4-2
Summary of Organic Method Calibrations
(continued)

Analysis	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP 88[3]	CLP 90[3]	OTHER
Petroleum Hydrocarbons, Total Recoverable, by IR (continued)	Ending	1 level Expected response should be within ± 15%	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Petroleum Hydrocarbons, Total, by GC	Initial	Not Applicable	Not Applicable	Not Applicable	Not Applicable	GRO[15] & DRO[14] Methods: Minimum of 5 levels, lowest near but above MDL. If % RSD[4] ≤ 25 use mean RF. Otherwise, plot calibration curve. --- Minimum of 3 levels RSD[4] ≤ 20% use mean CF. Otherwise, if RSD ≤ 30% plot calibration curve
	Continuing	Not Applicable	Not Applicable	Not Applicable	Not Applicable	GRO[15] & DRO[14]: Mid-point QC check standard analyzed each working day. If not within ± 25% of predicted response, recalibrate. --- Single point for each analyte at the beginning of each sequence or 24 hours ≤ 15% D[5] from initial for each analyte --- ≤ 10% D[5] for California LUFT
	Ending	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Run to confirm system in control

Table 8.4-2
Summary of Organic Method Calibrations
(continued)

Analysis	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP 88[3]	CLP 90[3]	OTHER
Petroleum Hydrocarbons, Total, by GC (continued)	Other	Not Applicable	Not Applicable	Not Applicable	Not Applicable	TN GRO[12] & DRO[14]: Retention time windows established over 72 hour period by injection of each method component three times. --- Retention time windows established over 72 hour period by injection of method standard 3 times.
Purgeable Halocarbons by GC	Initial	Minimum of 3 levels, lowest near but above MDL. If % RSD[4] < 10, assume linearity and average RF is used. Otherwise, plot calibration curve.	Minimum of 5 levels, lowest near but above MDL. If % RSD[4] < 20, linearity is assumed and average RF is used. Otherwise, plot calibration curve.	Not Applicable	Not Applicable	5 levels If RSD[4] ≤ 20%, assume linearity. If > 20%, plot calibration curve.
	Continuing	1 or more calibration standards analyzed daily. Compare results to method calibration tables.	Mid-level calibration standard analyzed every 10 samples. If not within ± 15 % of predicted response, recalibrate.	Not Applicable	Not Applicable	1 every 10 samples, % D[5] ≤ 15% --- 1 every 10 samples, % D[5] ≤ 10% (≤ 15% for surrogates)
	Ending	Not Applicable	Retention times in windows for all compounds	Not Applicable	Not Applicable	Not Applicable
	Other	When doubt exists in compound identification, second column confirmation is recommended.	When doubt exists in compound identification, second column confirmation is recommended. Retention time windows established over 72-hour period by injection of each compound three times.	Not Applicable	Not Applicable	Not Applicable

Table 8.4-2
Summary of Organic Method Calibrations
(continued)

Analysis	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP 88[3]	CLP 90[3]	OTHER
Semivolatiles by GC/MS	Initial	Minimum of 3 levels, lowest near but above MDL If % RSD[4] ≤ 35%, can use mean RF[9]	Minimum of 5 levels, lowest near but above MDL. ≤ 30% RSD[4] for CCCs[18] SPCCs[17]: RF[9] ≥ 0.050	5 levels plus blank ≤ 30% RSD[4] for CCCs[18] SPCCs: RF_{avg}[9] or $RF_{daily} \geq 0.050$	5 levels (except 8 compounds which require 4 levels) ≤ 20.5 % RSD[4] of method Minimum RRF[10] of method	Not Applicable
	Continuing	1 level every 24 hours If not within ± 20% of predicted response, recalibrate	Mid-level standard every 12 hours after tuning < 30% RSD[4] for CCCs[18] SPCCs[17]: RF[9] ≥ 0.050	1 level every 12 hours after tuning ≤ 25% D[5] for CCCs[18] SPCCs[17]: RF_{avg}[9] or $RF_{daily} \geq 0.050$	1 mid-level standard every 12 hours after tuning ≤ 25 % D[5] of method Minimum RRF[10] and maximum RSD[4] of method	Not Applicable
	Ending	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Other	DFTPP[19] tuning every 24 hours before standard or sample runs. Check column performance daily with benzidine and pentachlorophenol	DFTPP[19] tuning at the beginning of every 12 hour shift.	DFTPP[19] tuning at the beginning of every 12 hour shift. SPCCs[17]: RF_{avg}[9] or $RF_{daily} \geq 0.050$	DFTPP[19] tuning at the beginning of every 12 hour shift for RT[21] and EICP area of internal standards evaluated against latest continuing calibration	Not Applicable
Semivolatiles by GC	Initial	Minimum of 3 levels, lowest near but above MDL. If % RSD[4] ≤ 10% use avg. RF. Otherwise plot calibration curve	Minimum of 5 levels, lowest near but above MDL. If % RSD[4] < 20 use average RF. Otherwise plot calibration curve.	Not Applicable	Not Applicable	Not Applicable

Table 8.4-2
Summary of Organic Method Calibrations
(continued)

Analysis	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP 88[3]	CLP 90[3]	OTHER
Semivolatiles by GC (continued)	Continuing	1 or more calibration standards analyzed daily. If not within ± 10 % of predicted response, recalibrate.	Mid-level calibration standard run every 10 samples. If not within ± 15% of predicted response, recalibrate	Not Applicable	Not Applicable	Not Applicable
	Ending	Not Applicable	Retennon times in windows for all compounds	Not Applicable	Not Applicable	Not Applicable
	Other	When doubt exists in compound identification, second column confirmation should be used.	When doubt exists in compound identification, second column confirmation should be used. Retention time windows established over 72 hour period by injection of each compound three times	Not Applicable	Not Applicable	Not Applicable
Volatiles by GC/MS	Initial	Minimum of 3 levels, lowest near but above MDL If % RSD[4] ≤ 35% can use mean RF[9]	Minimum of 5 levels, lowest near but above MDL % RSD[4] for CCCs[18] ≤ 30 SPCCs[17]: RF[9] ≥ 0.300 (0.250 for bromoform)	5 levels ≤ 30% RSD[4] of CCCs[18] SPCCs RF ≥ 0.300 (0.250 for Bromoform)	5 levels ≤ 20.5 % RSD[4] of method Minimum RRF[10] and maximum RSD must meet method criteria	Method 524.2: 5 levels plus blank ≤ 20% RSD[4] for all compounds ——— See operation-specific SOPs for air analysis
	Continuing	1 level every 24 hours after tune check standard Recoveries must meet limits given in method	Mid-level every 12 hours after tune check standard RF[9] of CCCs[18] ≤ 25% D[5] SPCCs[17]: RF[9] ≥ 0.300 (0.250 for bromoform)	1 level every 12 hours after tune check standard ≤ 25% D[5] for CCCs[18] SPCCs RF ≥ 0.300 (0.250 for Bromoform)	Mid-level every 12 hours after tune check standard %D[5] must be ≤ 25 from initial calibration Minimum RRF[10] must be met.	Method 524.2: 1 level every 8 hours after tune check standard ≤ 30% RSD[4] for all compounds ——— See operation-specific SOPs for air analysis
	Ending	Not Applicable	Not Applicable	Not Applicable	Not Applicable	See operation-specific SOPs for air analysis

Table 8.4-2
Summary of Organic Method Calibrations
(continued)

Analysis	Calibration	NPDES[1]	RCRA(SW846)[2]	CLP 88[3]	CLP 90[3]	OTHER
Volatiles by GC/MS (continued)	Other	BFB[20] tuning at the beginning of every 24 hour shift.	BFB[20] tuning at the beginning of every 12 hour shift.	BFB[20] tuning at the beginning of every 12 hour shift. SPCCs[17]: RF_{avg} or $RF^{[9]}_{daily} \geq$ 0.300 (0.250 for bromoform)	BFB[20] tuning at the beginning of every 12 hour shift for RT[21] and EICP area of internal standards evaluated against latest continuing calibration	Method 524.2: BFB[20] tuning at beginning of every 8 hour shift --- See operation-specific SOPs for air analysis
Volatiles by GC (Detectors in series)	Initial	Not Applicable	Minimum of 5 levels, lowest near but above MDL. If % RSD[4] ≤ 20, linearity assumed and average RF used. Otherwise, plot calibration curve.	Not Applicable	Not Applicable	Minimum of 3 levels, lowest near but above MDL. If % RSD[4] ≤ 10, linearity assumed and average RF[9] used.
	Continuing	Not Applicable	Mid-level calibration standard run every 10 samples. If not within ± 15% of predicted response, recalibrate. Retention times in windows for all continuing standards.	Not Applicable	Not Applicable	1 level every 10 samples % D[5] ± 15% from initial calibration
	Ending	Not Applicable	Retention times in windows for all compounds.	Not Applicable	Not Applicable	Not Applicable
	Other	Not Applicable	When doubt exists in compound identification, second column confirmation is recommended. Retention time windows established over 72 hour period by injection of each compound three times	Not Applicable	Not Applicable	When doubt exists in compound identification, second column confirmation is recommended. Retention time windows established over 72 hour period by injection of each compound three times

Table 8.4-2
Summary of Organic Method Calibrations
(continued)

(1) National Pollutant Discharge Elimination System
(2) Resource Conservation and Recovery Act (Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd edition, Final Update I, July 1992
(3) Contract Laboratory Program
(4) RSD - Relative Standard Deviation
(5) % D - Percent Difference
(6) TCDD - 2,3,7,8-Tetrachlorodibenzo-p-dioxin
(7) HRGC/LRMS - High Resolution Gas Chromatography/Low Resolution Mass Spectrometry
(8) HRGC/HRMS - High Resolution Gas Chromatography/High Resolution Mass Spectrometry
(9) RF - Response Factor
(10) RRF - Relative Response Factor
(11) DDT - Dichloro-diphenyl-trichloro-ethane
(12) CF - Calibration Factor
(13) PEM - Performance Evaluation Mixture
(14) DRO - Diesel Range Organics. DRO corresponds to alkane range of C_{10} - C_{24}.
(15) GRO - Gasoline Range Organics. GRO corresponds to an alkane range of C_6 - C_{10}.
(16) CRQL - Contract Required Quantitation Limit
(17) SPCC - System Performance Check Compound
(18) CCC - Continuing Calibration Compounds
(19) DFTPP - Decafluorotriphenylphosphine
(20) BFB - Bromofluorobenzene
(21) RT - Retention Time

TABLE 8.4-3
Periodic Equipment Calibrations

Type of Calibration	Calibration Requirements
Balance Calibration	• Must be serviced and calibrated annually by a manufacturer's representative • Calibration must be checked daily before use by analyst with weight(s) classified as Class "S" (or Class "S" traceable) by NIST per operation-specific SOPs. • Quarterly calibrations must cycle through the range of weights applicable to a balance as described in operation-specific SOPs. • All Class "S" weights must be certified by an outside agency every three years. • Class "S" traceable weights used for daily balance calibration checks must be checked against a Class "S" weight monthly.
Thermometer Calibration	• Working thermometers must be calibrated against a certified NIST thermometer at least annually as described in operation-specific SOPs. • The NIST thermometer must be re-certified every three years.
Micropipettors	• Calibrations are checked gravimetrically as required by the operation-specific SOP. • Must be calibrated at the frequency (normally quarterly) required by the manufacturer at a minimum.
Syringes, Volumetric Glassware and Graduated Glassware	• All syringes and volumetric glassware are purchased as Class A items. • Class A items are certified by the manufacturer to be within ± 1% of the measured volume, therefore, calibration of these items by ITAS-laboratories is not required. • All analysts are trained in the proper use and maintenance of measuring devices to ensure the measurement of standards, reagents and sample volumes are within method tolerances.

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Acidity	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable
Alkalinity	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Alkalinity (continued)	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable
Ammonia	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Ammonia (continued)	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within laboratory control chart limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: ≤ 20 % RPD[4] limit Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable
Ammonia (TKN)	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Ammonia (TKN) (continued)	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable
BOD	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable		

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Bromide	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples (or 1 per 10 samples by IC) Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples (or 1 per samples 10 by IC) Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.
Canon Exchange Capacity	See ICAP Procedures, this is an extraction method.				

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Chemical Oxygen Demand (COD)	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable
Chloride	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable

INTERNATIONAL TECHNOLOGY CORPORATION

Procedure Change No.:

OSOAMP-05

IT ANALYTICAL SERVICES DIVISION PROCEDURE/DOCUMENT CHANGE

PROCEDURE/DOCUMENT NUMBER: N/A			
PROCEDURE/DOCUMENT TITLE: ITAS Operation-Specific Quality Assurance Management Plan (OS QAMP) (Revision 0, September 1, 1993)			
PROCEDURE/DOCUMENT SECTION(S) AFFECTED BY CHANGE: Table 8.4-1, "Summary of Inorganic Method Calibrations", page 161.			
REASON FOR ADDITION OR CHANGE: Addition to Hydrazine (colorimetric) to the table.			
CHANGE EFFECTIVE FROM:	02/18/94	TO:	Ongoing
SAMPLES OR PROJECTS AFFECTED: N/A			
CHANGE: Add the attached table containing the requirements for hydrazine in front of page 161.			
SUBMITTED BY/DATE: Nasreen DeRubeis 02/07/94			
APPROVED BY:			
[signature] 2-22-94		TECHNICAL SPECIALIST/DATE	
[signature] 2/16/94		ITAS DIRECTOR, HEALTH & SAFETY/DATE	
[signature] 2/17/94		ITAS DIRECTOR, QA/QC/DATE	

INTERNATIONAL TECHNOLOGY CORPORATION

Procedure Change No.:

OSQAMP-06

ADDITION TO:
TABLE 8.5-1
Inorganic Laboratory Quality Control Samples

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Hydrazine Colorimetric	Method Blank	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples
	Matrix Spike Sample	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ± 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Frequency: 1 per batch of 20 samples Criteria: ± 20 % RPD[4] Corrective Action: Flag data outside of limit.

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Chloride (continued)	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples (or 1 per 10 samples by IC) Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Frequency: 1 per batch of ≤ 10 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples (or 1 per 10 samples for IC) Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Frequency: 1 per batch of ≤ 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable
Chlorine, Residual	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Chlorine, Residual (continued)	Matrix Spike Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Water	Not Applicable	Not Applicable
Chromium (Hexavalent) Cr^{+6}	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Frequency: 1 per batch of ≤ 20 samples prepped Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Frequency: 1 per batch of ≤ 20 samples prepped Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Frequency: 1 per batch of ≤ 20 samples prepped Criteria: percent recovery of analyte must be within laboratory control chart limits Corrective Action: Rerun all samples associated with unacceptable LCS	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Chromium (Hexavalent) Cr^{+6} (continued)	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within laboratory-established QC limits Corrective Action: Flag data outside of limit	Frequency: 1 per analytical batch of 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable matrix spike sample	Not Applicable	Frequency: 1 per batch of 20 samples Criteria: Must be within laboratory-established QC limits Corrective Action: Flag data outside of limit
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Frequency: 1 per analytical batch of 20 samples Criteria: ≤ 20 %RPD[4] limit Corrective Action: Reanalyze if sample remaining. If not, flag data outside of limit.	Not Applicable	Frequency: 1 per analytical batch of 20 samples Criteria: ≤ 20 %RPD[4] limit Corrective Action: Reanalyze if sample remaining. If not, flag data outside of limit.
Color	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Color (continued)	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable
Conductivity	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Cyanide (Amenable)	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Frequency: 1 per batch of ≤ 20 samples Criteria: percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS	Not Applicable	Not Applicable
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Frequency: 1 per analytical batch of 20 samples Criteria:Advisory limits are 75% - 125% recovery Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Frequency: 1 per analytical batch of 20 samples Criteria:Advisory limits are 75% - 125% recovery Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Cyanide (Amenable) (continued)	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable
Cyanide (Free)	Method Blank	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per batch of ≤ 20 samples prepped Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per batch of ≤ 20 samples prepped Criteria: percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike Sample	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per analytical batch of 20 samples Criteria: Limits are 75% - 125% recovery Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable matrix spike sample
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Cyanide (Free) (continued)	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per analytical batch of 20 samples Criteria: Limits are ≤ 20% RPD[4] Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable duplicate sample
Cyanide (Total)	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Frequency: 1 per batch of ≤ 20 samples Criteria: % recovery must be within ± 20% Corrective Action: Rerun all samples associated with unacceptable LCS	Frequency: 1 per batch of ≤ 20 samples Criteria: percent recovery of analyte must be within 80% - 120% Corrective Action: Rerun all samples associated with unacceptable LCS	Not Applicable
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Frequency: 1 per analytical batch of 20 samples Criteria: Advisory limit is 75% - 125% recovery Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Flag data associated data. Post digestion spike if sample result ≥ 4 times the spike level.	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Cyanide (Total) (continued)	Matrix Spike Duplicate Sample	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: Limit is 75% - 125% recovery Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: Advisory limits are ≤ 20% RPD[4] Corrective Action: Flag data associated with unacceptable duplicate sample	Not Applicable
Flashpoint (Ignitability)	Method Blank	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Not Applicable	Frequency: 1 per batch of ≤20 samples Criteria: RPD[4] must be ≤ 20% Corrective Action: Flag data associated with unacceptable duplicate sample	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Fluoride	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Frequency: 1 per batch of ≤ 20 samples (or 1 per 10 samples by IC) Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of ≤ 20 samples (or 1 per 10 samples by IC) Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable
Hardness	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Hardness (continued)	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable
Iodide	Method Blank	Frequency: 1 per batch of 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Iodide (continued)	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable
Methylene Blue Active Substances (MBAS)	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit
	Matrix Spike Duplicate	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Methylene Blue Active Substances (MBAS) (continued)	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.
Nitrogen, Nitrate-Nitrite	Method Blank	Frequency: 1 per batch of 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Nitrate only: Frequency: 1 per batch of 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Frequency: 1 per batch of 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples (or 1 per 10 samples for IC) Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples (or 1 per 10 samples for IC) Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Odor	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable
Oil and Grease	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Frequency: 1 per batch of ≤ 20 samples Criteria: percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: percent recovery of analyte must be between 80 - 120 % Corrective Action: Rerun all samples associated with unacceptable LCS

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Oil and Grease (continued)	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Frequency: 1 per batch of 20 samples Criteria: Limits are 75% - 125% recovery Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable matrix spike sample
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample (Requires that two samples be sent by client)	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: Advisory limits are ≤ 20% RPD[4] Corrective Action: Flag data associated with unacceptable duplicate sample	Not Applicable	Frequency: 1 per analytical batch of 20 samples Criteria: Advisory limits are ≤ 20% RPD[4] Corrective Action: Flag data associated with unacceptable duplicate sample
pH	Method Blank	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: ≤ 20 % RPD[4] limit Corrective Action: Flag data outside of limit.	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: Advisory limits are ≤ 20% RPD[4] Corrective Action: Flag data associated with unacceptable duplicate sample	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: Advisory limits are ≤ 20% RPD[4] Corrective Action: Flag data associated with unacceptable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Phenolics	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Frequency: 1 per batch of ≤ 20 samples Criteria: percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS	Not Applicable	Not Applicable
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Frequency: 1 per analytical batch of 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Frequency: 1 per batch of 20 samples Criteria: Limits are 75% - 125% recovery Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Phosphorus (Total and Ortho-phosphate)	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples (or 1 per 10 samples for IC) Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples (or 1 per 10 samples for IC ortho-phosphate determination only) Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Silica	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.
Solids	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Solids (continued)	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Reanalyze if sample remaining. If not, flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable
Sulfate	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: Rerun all samples associated with unacceptable LCS (ICV)	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: Rerun all samples associated with unacceptable LCS (ICV)

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Sulfate (continued)	Matrix Spike Sample	Frequency: 1 per batch of ≤ 20 samples (or 1 per 10 samples by IC) Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Frequency: 1 per batch of 20 samples Criteria: Limits are 75% - 125% recovery Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable matrix spike sample	Not Applicable	Frequency: 1 per batch of 20 samples Criteria: Limits are 75% - 125% recovery Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable matrix spike sample
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of ≤ 20 samples (or 1 per 10 samples by IC Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Frequency: 1 per analytical batch of 20 samples Criteria: ≤ 20 %RPD[4] limit Corrective Action: Reanalyze if sample remaining. If not, flag data outside of limit.	Not Applicable	Frequency: 1 per analytical batch of 20 samples Criteria: ≤ 20 %RPD[4] limit Corrective Action: Reanalyze if sample remaining. If not, flag data outside of limit.
Sulfide	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Sulfide (continued)	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable
Sulfite	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Sulfite (continued)	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable
Total Organic Carbon (TOC)	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Frequency: 1 per batch of ≤ 20 samples Criteria: percent recovery must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS	Not Applicable	Frequency: 1 per batch of 20 samples Criteria: Must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Must be within QC limits Corrective Action: Flag data outside of limit	Frequency: 1 per batch of 20 samples Criteria: Limits are 75% - 125% recovery Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable matrix spike sample	Not Applicable	Frequency: 1 per batch of 20 samples Criteria: Must be within laboratory control chart limits Corrective Action: Flag data outside of limit
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Total Organic Carbon (TOC) (continued)	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Frequency: 1 per analytical batch of 20 samples Criteria: ≤ 20 %RPD[4] limit Corrective Action: Reanalyze if sample remaining. If not, flag data outside of limit.	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: ≤ 20 % RPD[4] limit Corrective Action: Flag data outside of limit.
Total Organic Halides (TOX)	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples prepped Criteria: percent recovery of analyte must be within ± 20% Corrective Action: Rerun all samples associated with unacceptable LCS	Frequency: 1 per batch of ≤ 20 samples prepped Criteria: percent recovery of analyte must be within ± 20% Corrective Action: Rerun all samples associated with unacceptable LCS	Not Applicable	Not Applicable
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Limits are 75% - 125% recovery Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable matrix spike sample	Frequency: 1 per batch of 20 samples Criteria: Limits are 75% - 125% recovery Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Total Organic Halides (TOX) (continued)	Duplicate Sample	Frequency: 1 per analytical batch of 20 samples Criteria: ≤ 20 %RPD[4] limit Corrective Action: Reanalyze if sample remaining. If not, flag data outside of limit.	Frequency: 1 per analytical batch of 20 samples Criteria: ≤ 20 %RPD[4] limit Corrective Action: Reanalyze if sample remaining. If not, flag data outside of limit.	Not Applicable	Not Applicable
Turbidity	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples Criteria: Percent recovery must be within laboratory control chart limits Corrective Action: If not within laboratory control chart limits, rerun all associated samples	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: ≤ 20 % RPD[4] Corrective Action: Flag data outside of limit.	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
GFAA and Flame AA Metals. Mercury by CVAA	Method Blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Frequency: 1 per batch of ≤ 20 samples Criteria: Concentration ≤ CRDL[4]. If not, all samples between the CRDL and 10 X the blank values must be redigested. See CLP method for details. Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤20 samples Criteria: percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS	Frequency: 1 per batch of ≤20 samples Criteria: percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS	Frequency: 1 per batch of ≤20 samples Criteria: percent recovery of analyte must be between 80-120% (or supplier's established range for solid) Corrective Action: Rerun all samples associated with unacceptable LCS	Not Applicable
	Matrix Spike Sample	Frequency: 1 per batch of 20 samples Criteria: Limits for percent recovery are 75 - 125% Corrective Action: Flag data associated with unacceptable MS. (See SOP for detailed corrective action procedure and for other QC procedures.)	Frequency: 1 per batch of 20 samples Criteria: Limits for percent recovery are 75 - 125% Corrective Action: Flag data associated with unacceptable MS. (See SOP for detailed corrective action procedure and for other QC procedures.)	Frequency: 1 per batch of ≤20 samples Criteria: Limits for percent recovery are 75 - 125% unless sample conc. is > 4X the true value of the matrix spike Corrective Action: Flag data associated with unacceptable MS unless sample conc. is > 4X the true value of the matrix spike	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
GFAA and Flame AA Metals, Mercury by CVAA (continued)	Matrix Spike Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: Limits for percent recovery are 75 - 125% Corrective Action: Flag data associated with unacceptable MSD	Frequency: 1 per batch of 20 samples Criteria: Limits for percent recovery are 75 - 125% Corrective Action: Flag data associated with unacceptable MSD Post digestion spike if sample result ≥ 4X the spike level. Serial dilution at 5X, %D[5] < 10	Not Applicable	Not Applicable
	Duplicate Sample	Not Applicable	1 per 20 samples	Frequency: 1 per analytical batch of ≤20 samples Criteria: Advisory limits of ≤20% RPD[4] Corrective Action: Flag data outside of range	Not Applicable
ICP Metals	Method Blank	Frequency: 1 per batch of ≤20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Frequency: 1 per batch of ≤20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	Frequency: 1 per batch of ≤20 samples Criteria: Concentration ≤ CRDL[6]. If not, all samples between the CRDL and 10 X the blank values must be redigested. See CLP method for details. Corrective Action: Rerun all samples associated with unacceptable blank	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
ICP Metals (continued)	Laboratory Control Sample	Frequency: 1 per batch of ≤20 samples Criteria: percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS	Frequency: 1 per batch of ≤20 samples Criteria: percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS	Frequency: 1 per batch of ≤20 samples Criteria: percent recovery of analyte must be between 80 - 120% except for Ag and Sb (or supplier's established range for solid) Corrective Action: Rerun all samples associated with unacceptable LCS	Not Applicable
	Matrix Spike Sample	Frequency: 1 per batch of ≤20 samples Criteria: Limits for percent recovery are 75 - 125% Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per batch of ≤20 samples Criteria: Limits for percent recovery are 75 - 125% Corrective Action: Flag data associated with unacceptable matrix spike sample Post digestion spike if sample result ≥ 4X the spike level. Serial dilution at 4X. %D[5] < 10	Frequency: 1 per batch of ≤20 samples Criteria: Advisory limits for percent recovery are 75 - 125% Corrective Action: Flag data associated with unacceptable matrix spike sample Post-digestion spike if sample result ≤ 4X the spike level	Not Applicable
	Matrix Spike Duplicate Sample	Frequency: 1 per batch of 20 samples Criteria: Limits for percent recovery are 75 - 125% Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per batch of 20 samples Criteria: Limits for percent recovery are 75 - 125% Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable

TABLE 8.5-1
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
ICP Metals (continued)	Duplicate Sample	Not Applicable	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: Limit is ≤ 20% RPD[4] Corrective Action: Flag data outside of range	Not Applicable

(1) National Pollutant Discharge Elimination System
(2) Resource Conservation and Recovery Act (Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd edition, Final Update I, July 1992
(3) Contract Laboratory Program
(4) RPD - Relative Percent Difference
(5) % D - Percent Difference
(6) CRDL - Contract Required Detection Limit

TABLE 8.5-2
Organic Laboratory Quality Control Samples

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Aromatic Volatiles by GC	Method Blank	Frequency: 1 per day Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is more frequent Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank	Not Applicable	1 per 10 samples
	Laboratory Control Sample	Frequency: 1 with each batch of samples or when new reagents are used Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS	Frequency: 1 per 20 samples or each batch of samples, whichever is more frequent Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS	Not Applicable	1 with MS/MSD (≤20 samples)
	Matrix Spike Sample	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	1 per batch (≤20 samples
	Matrix Spike Duplicate Sample	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike	Not Applicable	1 per batch of ≤ 20 samples

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Aromatic Volatiles by GC (continued)	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Surrogates	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: samples which have any surrogate outside of lab established control limits will be flagged as estimated. Upon client request will be reanalyzed once and both sets of data reported.	Not Applicable	All samples, standards, and blanks
	Internal Standards	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.	Not Applicable	Not Applicable
Dioxins/ Dibenzofurans	Method Blank	Frequency: 1 per batch of ≤ 20 samples extracted Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all positive samples associated with unacceptable blank	Frequency: 1 per batch of ≤ 20 samples extracted Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all positive samples associated with unacceptable blank	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples extracted Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS	Frequency: 1 per batch of ≤ 20 samples extracted Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS	Not Applicable	Not Applicable

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Dioxins/ Dibenzofurans (continued)	Matrix Spike Sample	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike duplicate sample	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike duplicate sample	Not Applicable	Not Applicable
	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Surrogates	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Internal Standards	Internal standards are added to all samples (QC samples included). Internal standard recovery should be between 40 % to 120 %	Internal standards are added to all samples (QC samples included). Internal standard recovery should be between 40 % - 120 % for Method 8280 and between 40 % - 135 % for Method 8290. Use limits in laboratory SOP.	Not Applicable	Not Applicable
Formaldehyde	Method Blank	Not Applicable	Not Applicable	Not Applicable	1 per 10 samples ≤ reporting limit
	Laboratory Control Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Formaldehyde (continued)	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Surrogates	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Internal Standards	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Herbicides	Method Blank	Frequency: 1 with each batch of samples	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is greater Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank. Re-extract if still unacceptable.	Not Applicable	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is greater Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank. Re-extract if still unacceptable.
	Laboratory Control Sample	Frequency: One with each batch of samples extracted or when new reagents used Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is greater Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	Not Applicable	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is greater Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Herbicides (continued)	Matrix Spike Sample	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within laboratory-established control limits. Corrective Action: Flag data associated with unacceptable matrix spike sample
	Matrix Spike Duplicate Sample	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable
	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within laboratory-established control limits. Corrective Action: Flag data associated with unacceptable duplicate sample

INTERNATIONAL TECHNOLOGY CORPORATION

Procedure Change No.:

OSQAMP-06

IT ANALYTICAL SERVICES DIVISION PROCEDURE/DOCUMENT CHANGE

PROCEDURE/DOCUMENT NUMBER: N/A			
PROCEDURE/DOCUMENT TITLE: ITAS Operation-Specific Quality Assurance Management Plan (OS QAMP) (Revision 0, September 1, 1993)			
PROCEDURE/DOCUMENT SECTION(S) AFFECTED BY CHANGE: Table 8.5-1, "Inorganic Laboratory Quality Control Samples", page 198.			
REASON FOR ADDITION OR CHANGE: Addition to Hydrazine (colorimetric) to the table.			
CHANGE EFFECTIVE FROM:	02/18/94	TO:	Ongoing
SAMPLES OR PROJECTS AFFECTED: N/A			
CHANGE: Add the attached table containing the requirements for hydrazine in front of page 198.			
SUBMITTED BY/DATE: Nasreen DeRubeis 02/07/94			
APPROVED BY:			
James H. Zahi 2-23-94		TECHNICAL SPECIALIST/DATE	
Brian E. Lepsdo 2/16/94		ITAS DIRECTOR, HEALTH & SAFETY/DATE	
[signature] 2/17/94		ITAS DIRECTOR, QA/QC/DATE	

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Herbicides (continued)	Surrogates	Recommended	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must fall within laboratory established control limits before sample analysis may proceed. Sample Criteria: samples which have any surrogate outside of control limits will be flagged.	Not Applicable	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: Surrogate control limit is 70 - 130 % recovery. If Blank fails, re-extract and reanalyze. If sample fails, reanalyze and report best result. If best result not within limits, report both results Sample Criteria: Samples which have any surrogate outside of control limits will be flagged.
	Internal Standards	Optional	Optional	Not Applicable	Not Applicable
Nitro-aromatics by HPLC	Method Blank	Not Applicable	Frequency: 1 per ≤ 20 samples or each extraction batch of samples, whichever is greater. Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank	Not Applicable	Frequency: 1 per batch of ≤ 20 samples extracted Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	Not Applicable	Frequency: 1 per ≤20 samples or each extraction batch, whichever is greater Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS	Not Applicable	Frequency: 1 per batch of ≤ 20 samples extracted Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Nitro-aromatics by HPLC (continued)	Matrix Spike Sample	Not Applicable	Frequency: 1 per batch of ≤ 20 samples received within a one month period Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: None. Matrix spike results will be submitted in final report when requested by client.
	Matrix Spike Duplicate Sample	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples received within a one month period Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: None. Matrix spike duplicate results will be submitted in final report when requested by client.
	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Surrogates	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Internal Standards	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Non-Methane Organic Compounds (NMOC)	Method Blank	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per day Criteria: All target analytes must be < reporting limit Corrective Action: Reanalyze, obtain acceptable blank before proceeding
	Laboratory Control Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Matrix Spike Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Non-Methane Organic Compounds (NMOC) (continued)	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per 20 samples Criteria: %D must be ≤ 30% Corrective Action: Flag data associated with unacceptable duplicate sample
	Surrogates	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Internal Standards	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PAHs by HPLC	Method Blank	Frequency: 1 with each batch of samples extracted or when new reagents used Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is more frequent Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 with each batch of samples extracted or when new reagents used Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS	Frequency: 1 per ≤ 20 samples or each extraction batch of samples, whichever is more frequent Criteria: Percent recovery must be within laboratory derived acceptance limits Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS	Not Applicable	Not Applicable

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
PAHs by HPLC (continued)	Matrix Spike Sample	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per analytical batch of ≤ 20 samples received within a one month period Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples received within a one month period Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable
	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Surrogates	Not specified in method	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: Results must fall within laboratory established control limits Sample Criteria: Samples which have any surrogate outside of control limits will be flagged.	Not Applicable	Not Applicable
	Internal Standards	Optional	Optional	Not Applicable	Not Applicable

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
PCBs by HRGC/ HRMS	Method Blank	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per 20 or fewer samples Criteria: No detects above lower calibration range Corrective Action: Discuss with client, track down potential sources of contamination
	Laboratory Control Sample	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per 20 or fewer samples Criteria: Percent recovery should be within 40 - 160 % Corrective Action: Verify calculations, track down potential sources of error, discuss with client
	Matrix Spike Sample	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per 20 or fewer samples Criteria: Percent recovery should be within 40 - 160 % Corrective Action: Verify calculations, track down potential sources of error, discuss with client
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per 20 or fewer samples Criteria: Percent recovery should be within 40 - 160 %, % Difference should be within ± 50 % Corrective Action: Verify calculations, track down potential sources of error, discuss with client
	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
PCBs by HRGC/ HRMS (continued)	Surrogates	Not Applicable	Not Applicable	Not Applicable	Surrogates spiked into method blank and all samples (QC included) Criteria: Percent recovery should be within 25 - 150 % Corrective Action: Verify calculations, track down potential sources of error, discuss with client
	Internal Standards	Not Applicable	Not Applicable	Not Applicable	Internal standards spiked into method blank and all samples (QC included) Criteria: Percent recovery should be within 25 - 150 % Corrective Action: Verify calculations, track down potential sources of error, discuss with client
Pesticides/ PCBs by GC	Method Blank	Frequency: 1 with each batch of samples extracted or when new reagents used Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is more frequent Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank	Frequency: 1 per case, 1 per 20 samples, or 1 per sample extraction batch, whichever is greater Criteria: All compounds < CRQL[4] Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank	Frequency: 1 per case, 1 per 20 samples, or 1 per sample extraction batch, whichever is greater Criteria: Conc. < reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Pesticides/ PCBs by GC (continued)	Laboratory Control Sample	Frequency: 1 with each batch of samples extracted or when new reagents used Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is greater Criteria: Percent recovery must be within laboratory derived acceptance limits Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS	Not Applicable	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is greater Criteria: percent recovery must be within laboratory-established control limits Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS
	Matrix Spike Sample	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 MS/MSD per sample delivery group or 1 per 20 samples, whichever is greater Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within laboratory-established control limits Corrective Action: Flag data associated with unacceptable matrix spike sample
	Matrix Spike Duplicate Sample	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 MS/MSD per sample delivery group or 1 per 20 samples, whichever is greater Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Pesticides/ PCBs[4] (continued)	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample
	Surrogates	Not specified in method	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: Results must fall within laboratory established control limits Sample Criteria: Samples which have any surrogate outside of control limits will be flagged.	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: percent recovery for surrogates should be within the advisory limits 60-150 % Sample Criteria: samples which have any surrogate outside of control limits will be flagged.	Not Applicable
	Internal Standards	Optional	Optional	Not Required	Not Applicable
Petroleum Hydrocarbons Total by IR	Method Blank	Frequency: 1 with each batch of samples extracted or when new reagents used Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank	Not Applicable	Not Applicable	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is greater Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Petroleum Hydrocarbons Total by IR (continued)	Laboratory Control Sample	Frequency: 1 with each batch of samples extracted or when new reagents used Criteria: percent recovery must be within laboratory established acceptance limits Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS	Not Applicable	Not Applicable	Frequency: 1 with each batch of samples extracted or when new reagents used Criteria: percent recovery must be within laboratory established acceptance limits Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS
	Matrix Spike Sample	Frequency: 1 per 20 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery must be within laboratory established acceptance limits Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within laboratory established acceptance limits Corrective Action: Flag data associated with unacceptable matrix spike sample
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within laboratory established acceptance limits Corrective Action: Flag data associated with unacceptable matrix spike sample
	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Surrogates	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Internal	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Petroleum Hydrocarbons (Total) by GC	Method Blank	Not Applicable	Not Applicable	Not Applicable	DRO[5] and GRO[6] Methods: Frequency: 1 per ≤20 samples or each extraction batch of samples, whichever is greater Criteria: Concentration less than reporting limit Corrective Action: Re-extract all samples associated with unacceptable blank
	Laboratory Control Sample	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per 20 samples Criteria: Percent recovery must be within control chart limits Corrective Action: Re-extract all samples associated with unacceptable LCS
	Matrix Spike Sample	Not Applicable	Not Applicable	Not Applicable	Recommended for specific sampling programs
	Matrix Spike Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample
	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Recommended for specific sampling programs

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Petroleum Hydrocarbons (Total) by GC (continued)	Surrogates	Not Applicable	Not Applicable	Not Applicable	Surrogates spiked into Method Blank and all samples (QC samples included) Method Blank and LCS Criteria: Recovery of surrogates should be within 50% - 150% Sample Criteria: Samples which have any surrogate outside of control limits will be flagged
	Internal Standards	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Purgeable Halocarbons by GC	Method Blank	Frequency: 1 per day Criteria: Concentration less than reporting limit Corrective Action: Reanalyze all samples associated with unacceptable blank	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is more frequent Criteria: Concentration less than reporting limit Corrective Action: Reanalyze all samples associated with unacceptable blank	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 with each batch of samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Reanalyze all samples associated with unacceptable LCS	Frequency: 1 per 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Reanalyze all samples associated with unacceptable LCS	Not Applicable	Not Applicable

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Purgeable Halocarbons by GC (continued)	Matrix Spike Sample	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable
	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Surrogates	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Reanalyze samples or flag sample data not meeting surrogate criteria	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: samples which have any surrogate outside of lab established control limits will be flagged as estimated. Upon client request will be reanalyzed once and both sets of data reported.	Not Applicable	Not Applicable

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Purgeable Halocarbons by GC (continued)	Internal Standards	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.	Not Applicable	Not Applicable
Semi-volatiles by GC/MS[7]	Method Blank	Frequency: 1 with each batch of samples extracted or when new reagents used Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is greater Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank	Frequency: 1 per batch of ≤ 20 samples extracted Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank	Frequency: 1 per batch of ≤ 20 samples extracted Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank
	Laboratory Control Sample	Frequency: 1 per batch of ≤ 20 samples extracted Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is greater Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS	Not Applicable	Frequency: 1 per batch of ≤ 20 samples extracted Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS
	Matrix Spike Sample	Frequency: 1 per 20 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is more frequent. Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Semi-volatiles by GC/MS[7] (continued)	Matrix Spike Duplicate Sample	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per analytical batch of ≤ 20 samples received within a 14 day period Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable
	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Surrogates	Surrogates spiked into method blank and all samples (QC included) Method Blank and LCS Criteria: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	Surrogates spiked into method blank and all samples (QC included) Method Blank and LCS Criteria: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Samples are allowed one acid and/or one base/neutral surrogate outside of control limits. If two or more acid or base/neutral surrogates are out of control limits or any one surrogate is less than 10% recovery the sample will be re-extracted and reanalyzed.	Not Applicable
	Internal Standards	Optional, may be used for quantitation.	Internal Standards are added to all samples (QC samples included). Internal standard area of daily standard must be within -50 % to +100 % from the last daily calibration check standard. Otherwise, sample is reanalyzed.	Internal Standards are added to the method blank and all samples (QC samples included). Internal standard areas must be within -50 % to +100 % from the last daily calibration check standard. Otherwise, sample is re-analyzed	Internal Standards are added to the method blank and all samples (QC samples included). Internal standard areas must be within -50 % to +100 % from the last daily calibration check standard. Otherwise, sample

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Semivolatiles by GC	Method Blank	Frequency: 1 with each batch of samples extracted or when new reagents used Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is more frequent Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank	Not Applicable	Not Applicable
	Laboratory Control Sample	Frequency: 1 with each batch of samples extracted or when new reagents used Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is greater Criteria: Percent recovery must be within laboratory derived acceptance limits Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS	Not Applicable	Not Applicable
	Matrix Spike Sample	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per analytical batch of ≤ 20 samples received within a one month period Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable
	Matrix Spike Duplicate Sample	Not Applicable	Frequency: 1 per analytical batch of ≤ 20 samples received within a one month period Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike	Not Applicable	Not Applicable

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Semivolatiles by GC (continued)	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Surrogates	Not specified in method	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: Results must fall within laboratory established control limits Sample Criteria: Samples which have any surrogate outside of control limits will be flagged.	Not Applicable	Not Applicable
	Internal Standards	Optional	Optional	Not Required	Not Applicable
Volatiles by GC/MS[7]	Method Blank	Frequency: 1 per day Criteria: Concentration less than reporting limit Corrective Action: Reanalyze all samples associated with unacceptable blank	Frequency: 1 per ≤ 20 samples Criteria: Concentration less than reporting limit Corrective Action: Reanalyze all samples associated with unacceptable blank	Frequency: 1 per 12 hours Criteria: Concentration less than reporting limit, except methylene chloride, acetone, 2-butanone must be ≤5X CRQL Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank	Frequency: 1 per 8 hours Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank
	Laboratory Control Sample	Frequency: Analyzed if MS results fall outside advisory limits Criteria: percent recovery for each analyte must be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	Frequency: 1 per 8 hours containing all analytes of concern Criteria: percent recovery must be within acceptable control chart limits Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Volatiles by GC/MS[7] (continued)	Matrix Spike Sample	Frequency: 1 per ≤ 20 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per 20 samples Criteria: No criteria specified in method
	Matrix Spike Duplicate Sample	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable
	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Frequency: 1 per 20 samples Criteria: % Difference within laboratory limits
	Surrogates	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Reanalyze samples, then flag sample data not meeting surrogate criteria	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Reanalyze samples, then flag sample data not meeting surrogate criteria	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria: All surrogates must be in control before sample analysis may proceed. Sample Criteria: CLP guidelines will be followed.	Surrogates spiked into Method Blank and all samples. Method Blank Criteria: All surrogates must be in control before sample analysis may proceed. Sample Criteria: If surrogates not in control, sample will be reanalyzed to prove/disprove matrix interference.

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Volatiles by GC/MS[7] (continued)	Internal Standards	Internal standards may be used for quantitation (optional).	Internal Standards are added to all samples (QC samples included). Internal standard area of daily standard must be within -50 % to +100 % from the last daily calibration check standard. Otherwise, sample is reanalyzed.	Internal Standards are added to the method blank and all samples (QC samples included). Internal standard areas must be within -50 % to +100 % from the last daily calibration check standard. Otherwise, sample is re-analyzed	Internal Standard added to all samples and QC samples
Volatiles by GC (Detectors in series)	Method Blank	Not Applicable	Frequency: 1 per 20 samples Criteria: Concentration less than reporting limit Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank	Not Applicable	Not Applicable
	Laboratory Control Sample	Not Applicable	Frequency: 1 per 20 samples or each extraction batch of samples, whichever is more frequent Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS	Not Applicable	Not Applicable
	Matrix Spike Sample	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable

TABLE 8.5-2
Organic Laboratory Quality Control Samples
(continued)

Analysis	QC Sample	NPDES[1]	RCRA(SW846)[2]	CLP[3] (88 and 90)	OTHER
Volatiles by GC (continued)	Matrix Spike Duplicate Sample	Not Applicable	Frequency: 1 per batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike sample	Not Applicable	Not Applicable
	Duplicate Sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Surrogates	Not Applicable	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: samples which have any surrogate outside of lab established control limits will be flagged as estimated. Upon client request will be reanalyzed once and both sets of data reported.	Not Applicable	Not Applicable
	Internal Standards	Not Applicable	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.	Not Applicable	Not Applicable

(1) National Pollutant Discharge Elimination System
(2) Resource Conservation and Recovery Act (Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd edition, Final Update I, July 1992
(3) Contract Laboratory Program
(4) CRQL - Contract Required Quantitation Limits
(5) DRO - Diesel Range Organics. DRO corresponds to alkane range of C_{10} - C_{24}.
(6) GRO - Gasoline Range Organics. GRO corresponds to an alkane range of C_6 - C_{10}.
(7) See operation-specific SOP for air analysis

Table 8.5-3
Field Quality Control Samples

Type	Applicability		Accuracy and Precision Application	Introduced By
	Inorganic	Organic		
Trip Blank (volatiles)		✓	Accuracy	Supplier of Containers
Field Blank	✓	✓	Accuracy	Field Sampler
Rinsate Blank	✓	✓	Accuracy	Field Sampler
Collocated Sample	✓	✓	Precision	Field Sampler
Replicated Sample	✓	✓	Precision	Field Sampler
Split Sample	✓	✓	Precision	Field Sampler
Field Duplicate	✓	✓	Precision	Field Sampler
Field Matrix Spike	✓	✓	Accuracy	Field Sampler

INTERNATIONAL TECHNOLOGY CORPORATION

Procedure Change No.:

OSQAMP-03

IT ANALYTICAL SERVICES DIVISION PROCEDURE/DOCUMENT CHANGE

PROCEDURE/DOCUMENT NUMBER: N/A			
PROCEDURE/DOCUMENT TITLE: ITAS Operation-Specific Quality Assurance Management Plan (OS QAMP) (Revision 0, September 1, 1993)			
PROCEDURE/DOCUMENT SECTION(S) AFFECTED BY CHANGE: Table 8.5-4, "Laboratory Quality Control Samples", page 241.			
REASON FOR ADDITION OR CHANGE: To update ITAS requirements for MS/MSDs and to reflect the new division SOP (ITAS-IT-QC-004) for LCSs.			
CHANGE EFFECTIVE FROM:	12/14/93	TO:	Ongoing
SAMPLES OR PROJECTS AFFECTED: N/A			
CHANGE: Replace page 241 with the attached page containing revised frequencies for matrix spikes (MS), matrix spike duplicates (MSD), and laboratory control samples (LCS).			
SUBMITTED BY/DATE: Patti Carswell 12/14/93			
APPROVED BY:			
Patti B. Carswell 12/14/93		TECHNICAL SPECIALIST/DATE	
Brian E. Lemcke 12/14/93		ITAS DIRECTOR, HEALTH & SAFETY/DATE	
Gaelle Hall 12/14/93		ITAS DIRECTOR, QA/QC/DATE	

Table 8.5-4
Laboratory Quality Control Samples

Type	Frequency	Applicability		Accuracy and Precision Application	Introduced By
		Inorganic/ Radiochemical	Organic		
Reagent Blank	Can be done as part of method blank, determine separately with each new batch of reagent/solvents.	✓	✓	Accuracy	Analyst
Duplicate	1 out of 20 or at least 1/month/run	✓	✓	Precision	Analyst/ Prep
Continuing Calibration Standard	With each group of samples	✓	✓	Accuracy	Analyst/ Prep
Surrogate Standard	All standards, method blanks, and samples		✓ Method Dependent	Accuracy	Analyst/ Prep
Matrix Spikes	1 per each group of 20 samples processed	✓	✓	Accuracy	Analyst/ Prep
Matrix Spike Duplicates	1 per each group of 20 samples processed	✓	✓	Both	Analyst/ Prep
Blank Spike (Laboratory Control Sample)	1 per prep batch	✓	✓	Accuracy	Analyst/ Prep
Analytical Spike	As specified in methods, or as needed	✓		Accuracy	Analyst/ Prep
Internal Standards	Each sample and standard	✓	✓	Both	Analyst/ Prep
Method Blank	With each group of samples, or beginning and end of each run. For GC/MS, method is spiked with surrogates 1 out of 10	✓	✓	Accuracy	Analyst/ Prep
Yield Monitors	Operation-specific	✓		Accuracy	Prep

TABLE 9.2-1
Laboratory Quality Control Samples/Measurements

Sample/Measurement	Purpose
Initial Calibration	Ensures the calculated concentration is within the limits of error as defined by the initial calibration conformance requirements. (For example by using an average calibration factor with a conformance limit of ≤ 20% relative standard deviation, all samples calculated will be within 20% of the true value.)
Continuing and Ending Calibration	Ensure that the calculated concentration is within the limits of error defined by the continuing calibration conformance criteria (*e.g.* all values are within 15% of the true concentration).
Solvent Blanks	Demonstrates that the solvent used to prepare standards and dilutions is free of interferences as regards a specific analysis.
Method Blanks	Demonstrate the laboratory systems (*e.g.* glassware cleaning procedures) and laboratory reagents used for the preparation and analysis of samples have not contributed to a false positive or negative measurement.
Reagent Blanks	Demonstrate that the reagents purchased by the laboratory are free of interferences as regards an analysis and do not contribute to false positive or false negative results.
Volatile Holding Blanks	Demonstrate that the volatile organic samples are not cross contaminated during storage at the laboratory.
Laboratory Control Sample	Demonstrates the laboratory's ability to perform an analysis within the conformance requirements of the method.

Table 8.5-4
Laboratory Quality Control Samples

Type	Frequency	Applicability		Accuracy and Precision Application	Introduced By
		Inorganic/ Radiochemical	Organic		
Reagent Blank	Can be done as part of method blank, determine separately with each new batch of reagent/solvents.	✓	✓	Accuracy	Analyst
Duplicate	1 out of 20 or at least 1/month/run	✓	✓	Precision	Analyst/ Prep
Continuing Calibration Standard	With each group of samples	✓	✓	Accuracy	Analyst/ Prep
Surrogate Standard	All standards, method blanks, and samples		✓ Method Dependent	Accuracy	Analyst/ Prep
Matrix Spikes	1 out of 20 or at least 1/month/run	✓	✓	Accuracy	Analyst/ Prep
Matrix Spike Duplicates	1 out of 20 or at least 1/batch	✓	✓	Both	Analyst/ Prep
Blank Spike (Laboratory Control Sample)	1 out of 20 or at least 1/batch with MS/MSD pair	✓	✓	Accuracy	Analyst/ Prep
Analytical Spike	As specified in methods, or as needed	✓		Accuracy	Analyst/ Prep
Internal Standards	Each sample and standard	✓	✓	Both	Analyst/ Prep
Method Blank	With each group of samples, or beginning and end of each run. For GC/MS, method is spiked with surrogates 1 out of 10	✓	✓	Accuracy	Analyst/ Prep
Yield Monitors	Operation-specific	✓		Accuracy	Prep

TABLE 9.2-1
Laboratory Quality Control Samples/Measurements

Sample/Measurement	Purpose
Initial Calibration	Ensures the calculated concentration is within the limits of error as defined by the initial calibration conformance requirements. (For example by using an average calibration factor with a conformance limit of ≤ 20% relative standard deviation, all samples calculated will be within 20% of the true value.)
Continuing and Ending Calibration	Ensure that the calculated concentration is within the limits of error defined by the continuing calibration conformance criteria (*e.g.* all values are within 15% of the true concentration).
Solvent Blanks	Demonstrates that the solvent used to prepare standards and dilutions is free of interferences as regards a specific analysis.
Method Blanks	Demonstrate the laboratory systems (*e.g.* glassware cleaning procedures) and laboratory reagents used for the preparation and analysis of samples have not contributed to a false positive or negative measurement.
Reagent Blanks	Demonstrate that the reagents purchased by the laboratory are free of interferences as regards an analysis and do not contribute to false positive or false negative results.
Volatile Holding Blanks	Demonstrate that the volatile organic samples are not cross contaminated during storage at the laboratory.
Laboratory Control Sample	Demonstrates the laboratory's ability to perform an analysis within the conformance requirements of the method.

INTERNATIONAL TECHNOLOGY CORPORATION

Procedure Change No.:

OSQAMP-01

IT ANALYTICAL SERVICES DIVISION
PROCEDURE/DOCUMENT CHANGE

PROCEDURE/DOCUMENT NUMBER: N/A			
PROCEDURE/DOCUMENT TITLE: ITAS Operation-Specific Quality Assurance Management Plan (OS QAMP) (Revision 0, September 1, 1993)			
PROCEDURE/DOCUMENT SECTION(S) AFFECTED BY CHANGE: Table 9.2-3, "Precision and Accuracy Measurements", page 244.			
REASON FOR ADDITION OR CHANGE: Typographical error in precision equation.			
CHANGE EFFECTIVE FROM:	12/8/93	TO:	Ongoing
SAMPLES OR PROJECTS AFFECTED: N/A			
CHANGE: Replace pages 243/244 (double-sided sheet) with the attached double-sided sheet containing the corrected equation on page 244 for precision calculations.			
SUBMITTED BY/DATE: Chris Rigell 12/08/93			
APPROVED BY:			
Patti B. Carswell 12/13/93		TECHNICAL SPECIALIST/DATE	
Brian I. Reynolds 12/13/93.		ITAS DIRECTOR, HEALTH & SAFETY/DATE	
[signature] 12/13/93		ITAS DIRECTOR, QA/QC/DATE	

TABLE 9.2-2
Matrix Quality Control Samples

Quality Control Sample	Purpose
Duplicate samples	Estimates the ability of the laboratory to obtain precise measurements on a sample. This measure is dependent on the homogeneity of the sample being duplicated. Solid samples often portray poor sample homogeneity and therefore often have poor duplication as regards to the sample result.
Matrix Spike Sample	Estimates the ability of the laboratory to obtain accurate measurements on a sample. The measure is dependent on the bias a sample matrix may cause regarding a given analyte.
Matrix Spike Duplicate Sample	In addition to verifying the accuracy of the matrix spike sample,the matrix spike duplicate can be used with the matrix spike sample as a measure of precision by calculating the relative percent difference (RPD).

TABLE 9.2-3
Precision and Accuracy Measurements

Measurement	Definition
Accuracy	The degree of agreement of a measurement with an accepted reference or true value. The only true or known values in the laboratory are spiked samples. Expressed as laboratory control sample percent recovery (LCS %R). $$LCS\ \%\ R = \frac{Found}{True} x\,100$$ Found = the concentration of an analyte determined from sample analysis. True = the concentration of the analyte spiked into the sample.
Precision	The measure of analytical reproducibility of two values. Expressed as the relative percent difference (RPD) of two values. $$RPD = \left[\frac{|x_1 - x_2|}{\left(\frac{x_1 + x_2}{2}\right)}\right] x\ 100$$ where: x_1 = first value x_2 = second value
Arithmetic mean $(\bar{x})$	The average of a set of values. $$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$ where: $\bar{x}$ = the mean x_i = the i^{th} data value n = number of data values

TABLE 9.2-2
Matrix Quality Control Samples

Quality Control Sample	Purpose
Duplicate samples	Estimates the ability of the laboratory to obtain precise measurements on a sample. This measure is dependent on the homogeneity of the sample being duplicated. Solid samples often portray poor sample homogeneity and therefore often have poor duplication as regards to the sample result.
Matrix Spike Sample	Estimates the ability of the laboratory to obtain accurate measurements on a sample. The measure is dependent on the bias a sample matrix may cause regarding a given analyte.
Matrix Spike Duplicate Sample	In addition to verifying the accuracy of the matrix spike sample, the matrix spike duplicate can be used with the matrix spike sample as a measure of precision by calculating the relative percent difference (RPD).

TABLE 9.2-3
Precision and Accuracy Measurements

Measurement	Definition
Accuracy	The degree of agreement of a measurement with an accepted reference or true value. The only true or known values in the laboratory are spiked samples. Expressed as laboratory control sample percent recovery (LCS %R). $$LCS\ \%\ R = \frac{Found}{True} x\,100$$ Found = the concentration of an analyte determined from sample analysis. True = the concentration of the analyte spiked into the sample.
Precision	The measure of analytical reproducibility of two values. Expressed as the relative percent difference (RPD) of two values. $$RPD = \left[\frac{|x_1 - x_2|}{\left(x_1 + \frac{x_2}{2}\right)}\right] x\ 100$$ where: x_1 = first value x_2 = second value
Arithmetic mean $(\bar{x})$	The average of a set of values. $$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$ where: $\bar{x}$ = the mean x_i = the i^{th} data value n = number of data values

TABLE 9.2-3
Precision and Accuracy Measurements
(Continued)

Measurement	Definition
Sample Standard Deviation (s)	A measure of the random (probable) error associated with a single measurement within a data set. $$s = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1}}$$ where: s = sample standard deviation $\bar{x}$ = the mean x_i = the i^{th} data value n = number of data values
Quality Control Chart	A graphical representation of analytical accuracy. Displays the arithmetic mean of a data set, the upper and lower warning limits and the upper and lower control limits.
Upper Control Limit (UCL)	$UCL = \bar{x} + 3s$
Upper Warning Limit (UWL)	$UWL = \bar{x} + 2s$
Lower Warning Limit (LWL)	$LWL = \bar{x} - 2s$
Lower Control Limit (LCL)	$LCL = \bar{x} - 3s$

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Sample Containers, Preservations, and Holding Times - Inorganic (continued)

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ITAS
Operation-Specific
Quality Assurance Management Plan

Appendix N

U. S. Navy-Specific Attachment

Approval: _____

Jack R. Hall
Division Director QA/QC

Date: December 1, 1993

This U.S. Navy-Specific Attachment contains statements of clarification and requirements which must be met on all Navy NEESA Projects unless stated otherwise in writing by the U.S. Navy.

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ITAS Operation-Specific Quality Assurance Management Plan
Appendix N
Navy-Specific Attachment

Section	Page	ITAS Clarification/Requirement
4.4.1	38	The Operation-Specific Quality Assurance Management Plan (OS QAMP) provides a definition of Type II water. This definition includes five specifications: conductivity, resistivity, total matter, color retention time, and soluble silica. **ITAS prepares reagent grade Type II water using commercial systems.** ITAS laboratories will perform and document daily checks of Type II water by monitoring conductivity and/or resistivity as recommended by the manufacturer. In addition to this daily check, method blanks are analyzed with each batch of samples prepped and/or analyzed. **Any contamination of the reagent water would be detected in the method blank analysis and appropriate corrective action will be taken.** Reagent grade water is further discussed in ITAS laboratory standard operating procedures (SOPs).
7.4	50	ITAS generates and follows SOPs that are derived from the methods they reference. The use of SOPs allows the laboratories to perform uniformly and to generate data of known quality. When the laboratory, for whatever reason, cannot follow the SOP within it's stated limits, a nonconformance memo is generated. The nonconformance memo describes the variance and any corrective action required. It must be signed by a supervisor and the corrective action must be documented and verified. **Any significant technical variances to the methods used on Navy projects that would have a technical impact on the resulting analytical data and that would affect a project will be submitted for approval by the Navy.**
8.5	59	The ITAS minimum requirement for QC samples is fifteen percent. For every twenty samples analyzed, three QC samples must be analyzed. The fifteen percent represents a percentage calculated based on the total QC samples divided by the total number of samples. This is the minimum and may be exceeded depending on regulatory programs or needs of the client. The tables in this section describe the actual regulatory program requirements. They break down the types of QC samples and indicate the minimum frequencies of each individual type of QC sample. For example, in Table 8.5-1 on page 183 for acidity, a method blank, an LCS, and a duplicate are required for every twenty client samples. While each individual QC sample is analyzed at a minimum rate of five percent, the three QC samples together along with twenty client samples equal a rate of fifteen percent.

ITAS Operation-Specific Quality Assurance Management Plan
Appendix N
Navy-Specific Attachment
(Continued)

Section	Page	ITAS Clarification/Requirement
8.5.1	59	The QC levels on page 59 (Section 8.5.1) were defined by ITAS several years ago. Levels I and II describe the minimum number and type of QC samples required. Level I was developed so that all client samples would fall under an acceptable quality assurance program even when no QC samples were required or requested by a client. In the past year, a new requirement for analyzing laboratory control samples (LCSs) has been developed for ITAS laboratories. The policy and procedure for LCSs are documented in an IT Analytical Services division SOP (ITAS-IT-QC-0004). In the next revision of the OS QAMP the definitions will be changed. **The last paragraph in Section 8.5 takes into account the new requirements for LCSs and lists the ITAS minimum QC samples.** This minimum requirement applies to methods that do not require specific QC samples. In the case where a client requests a method of this type, the laboratory would analyze the stated QC samples at a minimum.
Table 8.2-1	128	The column labeled "Other" in the tables describes the basic QA/QC program\requirements that ITAS will follow in the case where the client requests a method that is not decisive or requires a test where there are no method requirements. For example, if a client requires an amenable cyanide analysis on a TCLP leachate, we would then use the "Other" column since that analysis does not cover that matrix. All of the information listed in the "Other" columns are based upon our collective experience as an analytical division. **To the best of our knowledge, the Navy work will not be performed using any of the "Other" columns.**
Table 8.2-1	141	The 38 day holding time for mercury in glass (and 13 days in plastic) is the new promulgated requirement in the July 1992 SW-846. It was first promulgated in 1986. When Method 7470 was revised, the holding time was changed to 28 days. However, it did not go through the proper review procedure before it was promulgated. Update I was then promulgated which states 38 days. In proposed Update II, the original 28 days was presented, but that update has not been promulgated. **However, if the Navy specifies a holding time of less than 38 days, IT will use that specified holding time.**
Table 8.2-2	152	In regards to the analysis of volatiles in preserved and unpreserved samples, the following explanation is presented. In 40 CFR Part 136, it is stated, "Sample receiving no pH adjustment must be analyzed within seven days of sampling." The shorter holding time (7 days) therefore applies only to samples that did not receive the acid addition and the longer holding time (14 days) applies only to samples that received the acid addition or pH adjustment. The tables use the terms "≤ 2" and "< 2". ITAS defines "≤ 2" and "< 2" as essentially the same thing since a sample with a pH of 1.99 and a sample with a pH of 2.00 would both be considered successfully preserved. **The differences in the OS QAMP tables are primarily due to the differences in the analytical methods and 40 CFR Part 136.**

ITAS Operation-Specific Quality Assurance Management Plan
Appendix N
Navy-Specific Attachment
(Continued)

Section	Page	ITAS Clarification/Requirement
Table 8.2-2	152	**The requirement for TCLP Leachate is 4° ± 2°C.**
Table 8.4-1	165	The ending calibration level acceptance criteria for sulfide is changed from ten percent to fifteen percent in order to maintain consistency with the ITAS minimum requirements.
Table 8.5-1	183-215	For the conventional chemistry analyses in these tables, ITAS laboratories will perform MS and MSD analyses.
Table 8.5-1	183-215	There appear to be inconsistencies in the frequency of QC samples in these Tables. Since the frequencies stated in the OS QAMP tables were taken out of the referenced methods, what we are seeing here is an inconsistency of the methods. In order to further define what is stated in the text, we offer this additional information: **Each group processed together (i.e. prep batch) will have an LCS and a Method Blank (MB). This group will be 20 or less samples. Each group of multiple batches that contains up to 20 samples will include an MS/MSD. A single prep batch of 20 samples would include a MB, LCS, MS, and MSD.** Also, many of the methods do not define the terminology, ITAS is in the process of generating a glossary of terms for inclusion in the OS QAMP. Prep batch, analytical batch, and QC batch are examples of terms that will be defined.
Table 8.5-1	198, 199	Inconsistent frequencies of QC samples for iodide are shown in these tables. **The minimum requirement of fifteen percent QC samples for iodide (as well as for all other analytes) will always be met.** In this case, ITAS defines "1 per batch of 20 samples" and "1 per batch of ≤ 20 samples" as the same thing since the minimum requirement will always be met.
Table 8.5-2	216, 238	The table shows an inconsistency for aromatic volatiles and volatiles by GC in the frequency of blank analyses. **ITAS will run a method blank with each group of samples processed.**
Table 8.5-2	235	To clarify the QC samples for semivolatiles by GC for RCRA, an MS and an MSD will be analyzed with every QC batch of 20 client samples. A method blank and a LCS will be analyzed with each prep batch of samples. The fifteen percent minimum QC samples will be exceeded. The current time limit to hold a QC batch open is one month.
Table 8.5-4	241	The OS QAMP is changed here to require a frequency of "1 MS and MSD per each group of 20 samples processed".
Table 8.5-4	241	The OS QAMP is changed here to require an LCS frequency of "1 per prep batch" to reflect the new division SOP (ITAS-IT-QC-004) for LCSs.

ITAS Operation-Specific Quality Assurance Management Plan
Appendix N
Navy-Specific Attachment
(Continued)

Section	Page	ITAS Clarification/Requirement
Miscellaneous Items		**Control Charting** The ITAS LCS Division SOP defines the standard requirement of LCSs for all analyses applicable. The spiked parameters (same parameters as MS/MSD) and if appropriate, surrogates will be control charted. Updating of control limits is required at least annually. The identification of out of control events is described along with corrective actions in the LCS SOP (ITAS-IT-QC-004). **Glassware Washing*** ITAS glassware washing procedures meet USEPA Requirements in USEPA-SW846 Revision 1, July, 1992 Chapter 3 (inorganic) and Chapter 4 (organic). Glassware washing procedures are described in laboratory SOPs. **Waste Disposal*** Waste disposal procedures are also described in laboratory SOPs. ITAS laboratories meet local, state, and federal requirements for waste disposal. **Data Flags** Data flags will be used for CLP methods. In other methods the data will be footnoted. **Second Column Confirmation** A second column confirmation of the qualitative presence of a compound found on the primary column will be performed on the GC analyses. **Resumes** Resumes of key laboratory personnel will be supplied with any new proposals or submittals by the Navy. Key personnel would include the Laboratory Director, Project Manager, Group Leaders, and the QC Coordinator at a minimum. *Submittals will be made by any ITAS Laboratory performing work for the Navy.

GLOSSARY

This glossary is intended to provide standard definitions for terms which are commonly used in ITAS laboratories.

Acceptance Limits
Data quality limits specified for analytical method performance.

Accuracy
Accuracy is a measure of the bias inherent in a system or the degree of agreement of a measurement with an accepted reference or true value. It is most frequently expressed as percent recovery. (See percent recovery).

Aliquot, Aliquant
A measured portion of a sample taken for analysis.

Analytical Batch
Same as QC Batch

Analytical Spike
A sample created by spiking target analytes into a prepared portion of a sample just prior to analysis. (Also see Matrix Spike.)

Arithmetic Mean (Also see Mean)
The arithmetic mean ($\bar{x}$) is the average of a set of values. It is equal to the sum of the observed values divided by the number of observations. Also called "average".

$$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$

where: $\bar{x}$ = the mean
x_i = the i^{th} data value
n = number of data values

Audit

A documented activity performed in accordance with a written program or checklists to verify, by examination and evaluation of objective evidence, that applicable elements of the quality assurance program have been developed, documented, and effectively implemented in accordance with specified requirements. An audit should not be confused with surveillance or inspection.

Bias

A systematic (consistent) error in test results. Bias is expressed as the difference between the population mean and the true or reference value, or as estimated from sample statistics, the difference between the sample average and the reference value.

Blind Performance Evaluation Sample

A sample either submitted to the laboratory or prepared in the laboratory whereby the concentrations of parameters of concern are known by the preparer and not by the laboratory.

Calibration

Establishment of a relationship between various calibration standards and the measurements of them obtained by a measurement system, or portions thereof. The levels of the calibration standard should bracket the range of levels at which actual measurements are to be made. Calibration is also the act of making a scheduled comparison of instrument performance against national standards for instruments which measure physical parameters such as mass, time, and temperature. This type of calibration is independent of use in specific analyses and projects.

Calibration Curve

The graphical relationship between the known values for a series of calibration standards and instrument responses.

Calibration Factor (CF) (Also see RF and RRF)

The ratio of the instrument response of an analyte to the amount injected. CFs are used in external standard calibrations.

$$CF = \frac{Total\ Area\ of\ Peak}{Mass\ Injected}$$

Calibration Standard
A standard used to quantitate the relationship between the output of a sensor and a property to be measured. Calibration standards should be traceable to Standard Reference Materials (provided by NIST, EPA, or other recognized standards agencies) or a primary standard.

Certificate of Analysis (COA)
The standard ITAS format for reporting analytical results.

Certified Reference Material (CRM)
A reference material accompanied by a certificate issued by an organization certifying the contents and concentration(s) of the material. (See also Standard Reference Material.)

Chain-of-Custody (COC)
A system of documentation demonstrating the physical custody and traceability of samples.

Check Standard Analyses
A standard (often a midpoint standard) analyzed at a frequency specified in the method or in an SOP to verify the continuing calibration of the standard curve.

Client Sample
The material or collection media submitted to the laboratory for analysis. Field QC samples are considered client samples but laboratory QC samples are not counted as client samples when counting samples for batches.

Coefficient of Variation (Relative Standard Deviation)
A measure of precision (relative dispersion). It is equal to the standard deviation (s) divided by the mean ($\bar{x}$) and multiplied by 100 to give a percentage value.

$$CV\ (RSD) = \left(\frac{s}{\bar{x}}\right) \times 100$$

Collocated Samples
Independent samples collected in such a manner that they are equally representative of the variable(s) of interest at a given point in space and time. The results will indicate sampling as well as analytical variability.

Comparability
A measure of the confidence with which one data set can be compared to another.

Completeness
The amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal operations. It is usually expressed as a percentage:

$$\%\ Completeness = \frac{V}{n} \times 100$$

where: V = number of measurements judged valid
n = total number of measurements

Composite
A sample composed of two or more increments.

Control Chart
A graphical representation of analytical accuracy. Displays the arithmetic mean of a data set, the upper and lower warning limits and the upper and lower control limits.

Control Table
A tabular presentation of test results with respect to time or sequence of measurement, together with limits within which the results are expected to lie when the analytical process is in a state of control.

Corrective Action
A measure taken to correct a deficiency, finding, or variance and to minimize the possibility of recurrence.

Correlation Coefficient
The correlation coefficient (r) is a determination of how closely data "fits" a straight line. It is a number between -1 and 1 that indicates the degree of linear relationship between two sets of numbers. A correlation coefficient of +1 (usually calculated to three decimal places or 1.000) means the data falls exactly on a straight line with positive slope. A correlation coefficient of -1 (or -1.000) means the data falls exactly on a straight line with negative slope.

Data Quality Objectives
The precision, accuracy, completeness and comparability goals to be achieved for a particular data set or project.

Data Validation (See Validation - Data)

Data Verification (See Verification - Data)

Deficiency
A deviation from accepted procedures, practices, or standards; or a defect in an item that is determined not to render the quality of an item or service unacceptable or indeterminate.

Degrees of Freedom
The number of independent deviations used in calculating an estimate of the standard deviation.

Double Blind Performance Evaluation Sample
A sample that contains select parameters at defined levels. The levels are unknown to the laboratory. The laboratory is also unaware that the sample is a performance evaluation sample.

Duplicate Sample Analyses
Different aliquots of the same sample are analyzed to evaluate the precision of an analysis.

Error
The difference between an observed or measured value and its true value.

Field Blank
A blank that is prepared and handled in the field and analyzed in the same manner as its corresponding client samples.

Field Matrix Spike
A sample created by spiking target analytes into a sample in the field at the point of sample acquisition.

Finding
An event discovered during an audit which, if continued, is sufficient to render the quality of an item unacceptable or indeterminate.

Geometric Mean
The n^{th} root of the product of all values in a set of n values or the antilogarithm of the arithmetic mean of the logarithms of all the values of a set of n values. The geometric mean is generally used when the logarithms of a set of values are nearly normally (Gaussian) distributed, such as is the case of much population data.

Initial Calibration
Analysis of a series of analytical standards at different specified concentrations; used to define the linearity and dynamic range of the response of an instrument to the target compounds prior to the analysis of samples.

Instrument Detection Limit (IDL)
The smallest concentration or amount an instrument can reliably detect.

Internal Standards (IS)
Compounds added to every standard, QC sample, client sample or sample extract at a known concentration prior to analysis for the purpose of quantitation. For example, internal standards are used as the basis for quantitation of the target compounds by GC/MS.

Linear Regression
A statistical method for finding a straight line that best fits a set of two or more data points, thus providing a relationship between two variables.

Manuals of Practice (MOP)
Detailed discussions of specific technical subjects. These documents provide detailed information relating to technical topics discussed in the Quality Assurance Management Plan. For example, a Manual of Practice for the field collection, preservation, and shipment of samples to ITAS laboratories provides specific uniform direction to IT associates.

Matrix
The component or substrate which contains the analyte(s) of interest. Examples of matrices are water, soil or sediment, and air. Matrix is not synonymous with phase (liquid or solid).

Matrix Effect
An interference in the measurement of analyte(s) in a sample that is caused by materials in the sample. Matrix effects may cause elevated reporting limits or may prevent the acquisition of acceptable results.

Matrix Spike (MS)
An aliquot of a matrix fortified (spiked) with known quantities of specific compounds and subjected to an entire analytical procedure in order to indicate the appropriateness of the method for a particular matrix. The percent recovery for the respective compound(s) is then calculated.

Matrix Spike Duplicate (MSD)
A second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

Mean (See Arithmetic Mean)

Measurement
The process or operation of ascertaining the extent, degree, quantity, dimensions, or capability with respect to a standard.

Median
The middle value of a set of data when the data set is ranked in increasing or decreasing order.

Method
An assemblage of techniques.

Method Blank
An analytical control consisting of all reagents, which may include internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background contamination. Examples of method blanks are a volume of deionized or distilled laboratory water for water samples, a purified solid matrix for soil/sediment samples, or a generated zero air.

Method Detection Limit (MDL)
The minimum concentration of an analyte that, in a given matrix and with a specific method, can be identified, measured, and reported with 99% confidence that the analyte concentration

is greater than zero. The MDL is operationally defined as:

$$MDL = st_{(n-1,\ \alpha=0.99)}$$

Where s = the standard deviation of a number of measurements of a blind or sample matrix containing the analyte at a concentration near the lowest standard recommended in the method and

$t_{(n-1,\ \alpha=0.99)}$ = the student's value for a one-sided t-statistic appropriate for the number of samples used to determine (s), at the 99% confidence level and n-1 degrees of freedom.

Modified Method
A standard or reference method which has been changed to meet project or matrix requirements.

Nonconformance
Any event which is beyond the limits established for laboratory operation. A variance in characteristic, documentation, or procedure which may be sufficient to render the quality of an item unacceptable or indeterminate.

Observation
An isolated instance of noncompliance or questionable practice. A situation that could become a finding if left unresolved.

Operation-Specific QAMP
See Quality Assurance Management Plan.

Operational Calibration
Routinely performed as part of instrument usage, such as the development of a standard calibration curve. Operational calibration is generally performed for instrument systems.

Outlier
A result excluded from the statistical calculations due to being deemed "suspicious" when applying the "Grubbs Test" (or equivalent).

Parameter
A constant or coefficient that describes some characteristic of a population (e.g., standard deviation, mean, regression coefficients). Also, a chemical being measured, i.e., an analyte.

Percent Difference
When two independent measurements of the same characteristics are available, it is possible to use the percent difference instead of the coefficient of variation to measure precision.

$$\%D = \left| \frac{X_1 - X_2}{X_1} \right| \times 100\%$$

where: %D = percent difference
X_1 = first value
X_2 = second value

Percent Recovery
A measure of accuracy determined from the comparison of a reported spike value to its true spike concentration.

$$\%R = \frac{observed\ conc. - sample\ conc.}{true\ spike\ conc.} \times 100\%$$

Performance Audit
Planned independent sample checks of actual output which are made on a random basis to arrive at a quantitative measure of the quality of the output. They are conducted on an ongoing basis within the laboratory by the Quality Assurance/Quality Control Coordinator and the ITAS Director of QA/QC. These audits are reported to the Laboratory Director.

Periodic Calibration
A calibration that is performed at prescribed intervals for equipment such as balances, thermometers, and balance weights. In general, they are performed on equipment that are distinct, singular purpose units, and are relatively stable in performance.

Population
A generic term denoting any finite or infinite collection of individual things, objects, or events.

Pre-Award Survey
On-site inspection, review, and discussions with prospective contractors. Discussions would normally include, but not be limited to, the proposed project plan, personnel, procedures, schedule, and facilities.

Precision
A measurement of mutual agreement (or variability) among individual measurements of the same property, usually under prescribed similar conditions. Precision is usually expressed in terms of relative standard deviation or relative percent difference, but can be expressed in terms of the variance, range, or other statistic.

Prep Batch
A group of client samples processed through a preparation procedure in the same time frame.

Preventive Maintenance
An organized program, within ITAS laboratories, of actions (such as equipment cleaning, lubricating, reconditioning, adjustment and/or testing) taken to maintain proper instrument and equipment performance and to prevent instruments and equipment from failing during use.

Primary Standard
A material having a known, stable property that can be accurately measured or derived from established physical or chemical constants. It is readily reproducible and can be accepted (within stated limits) and used to establish the same value of another substance or item.

Procedure
Detailed instructions to permit replication of a method. (See Standard Operating Procedure.)

Proficiency Testing
Special series of planned tests which will determine the ability of field technicians or laboratory analysts to perform routine analyses. The results from this testing may be used for comparison against established criteria or for relative comparisons among the data from a group of technicians or analysts.

Project-Specific Manual
A manual that describes analytical and/or QA procedures required by a regulatory agency or by contract. It may supplement or change Quality Assurance and/or Quality Control practices for a specific project.

Protocol
Methodology specified in regulatory, authoritative, or contractual situations.

QC Batch
A group of prep batches totaling up to 20 client samples for a similar analysis of the same matrix type, plus all the necessary QC samples (minimum QC is Blank, LCS, MS and MSD).

QC Check Sample
A reference matrix containing known concentrations of parameters of interest. If prepared in the laboratory, it is made using stock standard solutions independent of those used for calibration. If the results of these parameters do not meet acceptance criteria, corrective actions are taken.

Qualification (Personnel)
The characteristics of abilities gained through education, training, or experience, as measured against established requirements, such as standards or tests, that qualify an individual to perform a required function.

Quality
The totality of feature and characteristics of a product or service that bears on its ability to satisfy a given purpose. Absence of defects. IT defines Quality as "meeting the requirements of our clients, both internal and external".

Quality Assurance (QA)
"All those planned systematic actions necessary to provide confidence that a product or service will satisfy given needs." ANSI/ASQC Standard A3

Quality Assurance Management Plan (QAMP)
An orderly assembly of management policies, objectives, principles, and general procedures by which ITAS outlines how it intends to produce quality data. The Quality Assurance Management Plan (QAMP) defines the ITAS Quality Assurance Program. The QAMP discusses all aspects for quality assurance and quality control, both administrative and technical. However, it is not intended that the QAMP provide in-depth technical discussion. The QAMP has precedence in policy matters over all other ITAS quality-related documents. The QAMP is supplemented and further defined by the Operation-Specific Quality Assurance Management Plan.

Quality Assurance Project Plan (QAPjP)
An orderly assembly of detailed and specific procedures by which an agency or laboratory delineates how it produces quality data for a specific project or measurement method.

Quality Control (QC)
The daily, specific actions taken within the laboratory to verify sample integrity, performance of analyses, data processing, and record maintenance. A system of inspections, testing, and remedial actions applied to a process or operation so that, by inspecting a small portion (a sample) of the product currently produced, an estimate can be made of its quality and whether or not changes need to be made to achieve or maintain a predetermined or required level of quality.

Random Error
Variations of repeated measurements that are random in nature and individually not predictable.

Range
The difference between the largest and smallest numbers in a set of numbers.

Raw Data
All documentation associated with the original recording of analytical results pertinent to a specific sample or set of samples. This may include laboratory worksheets, calculation forms, instrument-generated output, analyst notes, etc., from sample receipt through final reporting.

Reagent Blank
Sample composed of materials (water, etc.) which will be analyzed along with client samples. If contaminants are found in the reagents at levels affecting these sample results, corrective actions must be taken. (Also see Method Blank.)

Reagent Water
Water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. ASTM Type II reagent water specifications are:

Maximum Total Matter (mg/l)	Maximum Electrical Conductivity at 25°C (mho/cm)	Minimum Electrical Resistivity at 25°C (M ohms/cm)	Minimum Color Retention Time of KMnO (min)
0.1	1.0	1.0	60

The reagent water's purity and acceptability is verified by analysis with each set of samples.

Recovery
See Percent Recovery

Reference Method
A method of known and demonstrated accuracy.

Regression Coefficients
The quantities describing the slope and intercept of a regression line.

Relative Error
An error expressed as a percentage of the true value or accepted reference value.

Relative Percent Different (RPD)
Statistic for evaluating the precision of a replicate set. For replicate results x_1 and x_2:

$$RPD = \frac{|x_1 - x_2|}{\left(\frac{x_1 + x_2}{2}\right)} \times 100\%$$

Relative Response Factor (RRF) (See also CF and RF)
A measure of the relative mass spectral response of a compound compared to its internal standard. RRFs are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. Because a RRF is the comparison of two responses, it is a unitless number. RRFs are determined by the following equation:

$$RRF = \frac{A_x}{A_{IS}} \times \frac{C_{IS}}{C_x}$$

Where: A = area of the characteristic ion measured
C = concentration
IS = internal standard
x = analyte of interest

Relative Standard Deviation
See Coefficient of Variation.

Replicates
Repeated but independent determinations of the same sample, at essentially the same time and under the same conditions.

Representative Sample
A sample taken to represent a lot or population as accurately and precisely as possible.

Representativeness
The degree to which a sample or group of samples is indicative of the population being studied.

Reproducibility
The precision, usually expressed as a standard deviation, measuring the variability among results of measurements of the same sample at different laboratories.

Response Factor (RF) (Also see CF and RRF)
A factor derived from the calibration of a compound that is used in the quantitation calculation of sample analytes. A response factor may be derived from an external standard calibration (then called a Calibration Factor) or from an internal standard calibration (then called a Relative Response Factor).

Secondary Standard
A material having a property that is calibrated against a primary standard.

Spiked Sample
A sample of material (gas, solid, or liquid) to which is added a known amount of some substance of interest.

Split Sample
A sample divided into two portions, one of which is sent to a different organization or laboratory and subjected to the same environmental conditions and steps in the measurement process as the one retained in-house.

Standard Addition
The procedure of adding known increments of the analyte of interest to a sample to cause increases in detection response to subsequently establish by extrapolation of the plotted responses the level of the analyte of interest present in the original sample.

Standard Deviation

A measure of the dispersion about the mean of the elements in a population. The square root of the variance of a set of values:

$$s = \sqrt{\frac{n\sum(X^2) - (\sum X)^2}{n(n-1)}}$$

where: s = standard deviation
$\sum$ = sum of
X = observed values
n = number of observations

Standard Method

A method of known and demonstrated precision issued by an organization generally recognized as competent to do so.

Standard Operating Procedures (SOP)

Written, detailed documents describing the performance of routine laboratory tasks. They specify what is done, whose responsibility it is to perform tasks and to verify their correctness. They are sufficiently detailed to provide data of known quality and integrity, with a minimum loss of data due to out-of-control situations. They also provide for documentation to record the performance of all tasks and their results, and they demonstrate the verification of the data each time the data are recorded, calculated, or transcribed. ITAS has two types of SOPs. Operating unit SOPs describe how to perform an operation specific to that unit and ITAS Division SOPs which supersede other SOPs and are applicable to all operating units.

Standard Reference Material (SRM)

A material produced in quantity, of which certain properties have been certified by the National Institute of Standards and Technology (NIST), formerly NBS, or other agencies to the extent possible to satisfy its intended use.

Standardization

The establishment of the value of a potential standard with respect to an established or known standard.

Statistic

A constant or coefficient that describes some characteristic of a sample. Statistics are used to estimate parameters of populations.

Stock Solution
A concentrated solution of analyte(s) or reagent(s) prepared and verified by prescribed procedure(s), and used for preparing working standards or standard solutions.

Subsample
A portion taken from a sample. A laboratory sample may be a subsample of a gross sample; similarly, a test portion may be a subsample of a laboratory sample.

Surrogates (Surrogate Standard)
Compounds, when required by a method, that are used added to every blank, sample, matrix spike, matrix spike duplicate, and standard. They are used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds that are not expected to be detected in environmental media.

Surveillance
The act of monitoring or observing to verify whether an item or activity conforms to specified requirements.

System Audit
A systematic on-site qualitative review of facilities, equipment, training, procedures, recordkeeping, data verification, and reporting aspects of a quality assurance system to arrive at a measure of the capability of the system. Within ITAS, system audits are performed on a periodic basis under the direction of the ITAS Director of QA/QC.

Systematic Error
The condition of a consistent deviation of the results of a measurement process from the reference or known level.

Technique
Physical or chemical principle for characterizing materials of chemical systems.

Traceability of Data
The entire documented chain of acquired data from the original acquisition effort through to the final tabulation, synthesis, reduction, and storage activities. The documentation will allow complete reconstruction of the data.

Traceability of Samples

During all environmental monitoring field efforts, acquired samples will be assigned specific and unique identification numbers. These sample numbers shall be accompanied by documentation (chain-of-custody form) which clearly identifies all parameters associated with sample acquisition. All additional sample numbering systems applied to the sample must be clearly cross-referenced to the field sample number to provide for traceability of samples from acquisition to reporting of sample results.

Traceability of Standards

The ability of an analytical standard material used for calibration purposes to be traced to its source. The standards used by ITAS must be traceable via written documentation to sources which produce or sell verified or certified standards, i.e., National Institute for Standards and Technology, USEPA, or vendors preparing standards from those sources which they have certified.

Trip Blank Analyses

Trip blanks are prepared by filling two VOA vials with organic-free water. They are shipped with each VOA field kit (group of samples). Trip blanks accompany the sample bottles through collection and shipment to the laboratory and are stored with the samples. If the trip blanks indicate possible contamination of the samples, depending upon the nature and extent of the contamination, the samples may either be corrected for the trip blank concentration or the sources re-sampled.

Validation - Computer Software

The process of establishing documented evidence which provides a high degree of assurance that software will consistently produce a product meeting its predetermined specifications and quality attributes. This process demonstrates that the mathematical or statistical model embodied in the computer program is an acceptable representation of the process for which it is intended and meets all specified requirements.

Validation - Data

The process of a second party performing a systematic review of the raw and final data produced by a laboratory using predetermined criteria to ascertain the validity of the data with respect to the criteria (e.g., HAZWRAP data validation).

Verification - Computer Software
The process of comparing software performance against known results to ensure that it correctly performs its intended function.

Verification - Data
The process of reviewing data to ensure that data reduction has been correctly performed and that analytical results to be reported correspond to the data acquired and processed.

Operation-Specific
QUALITY ASSURANCE MANAGEMENT PLAN
IT ANALYTICAL SERVICES DIVISION

KNOXVILLE LABORATORY
APPENDIX

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APPENDIX

KNOXVILLE - 0

LISTS OF APPENDIX SECTIONS, TABLES, AND FIGURES

LIST OF APPENDIX SECTIONS

LIST OF FIGURES

LIST OF TABLES

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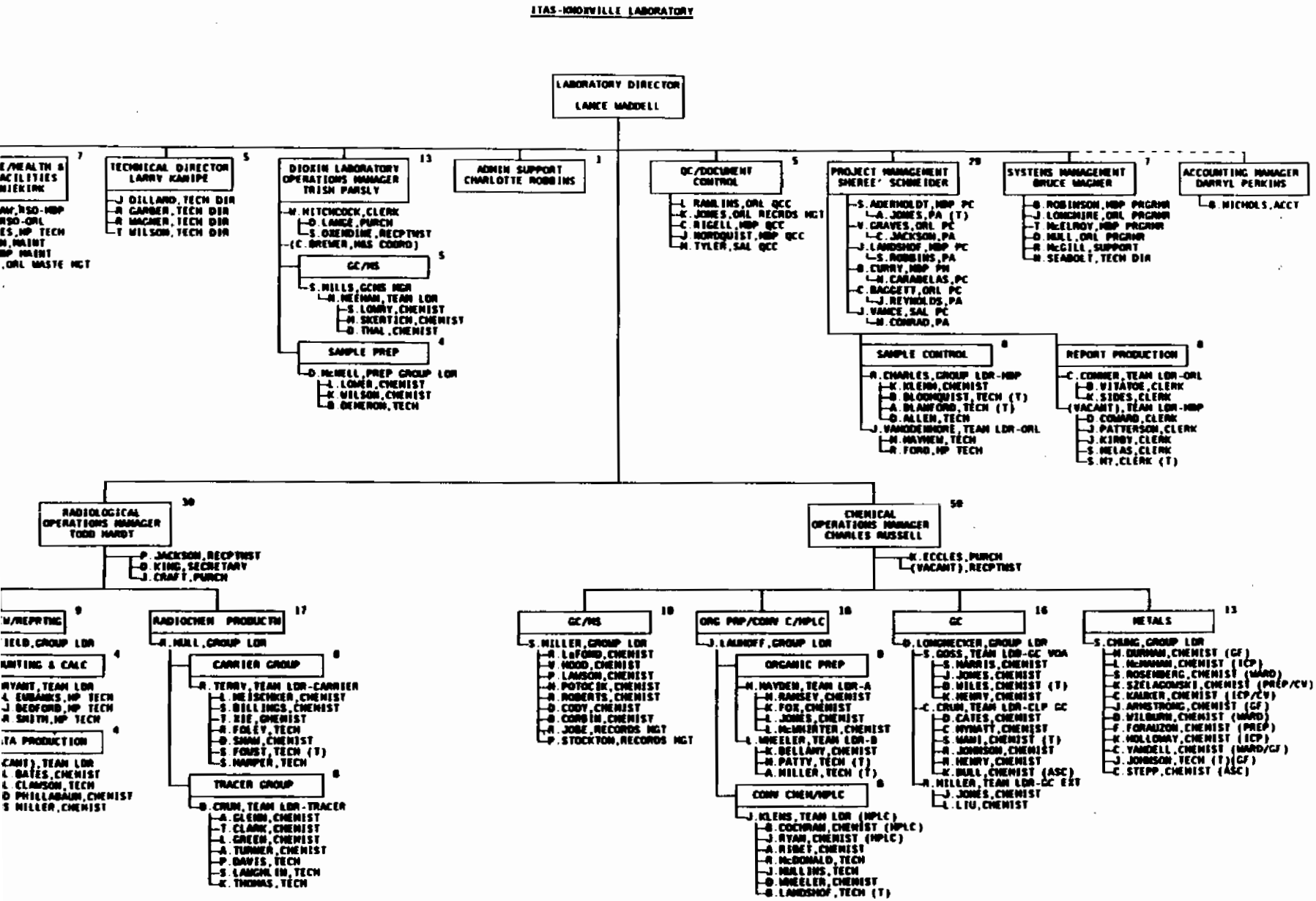
(continued)

APPENDIX

KNOXVILLE - 1

ORGANIZATIONAL CHART

FIGURE KN-1-1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LARRY KANIPE
J DILLARD, TECH DIR
R GARNER, TECH DIR
R WAGNER, TECH DIR
T WILSON, TECH DIR
DIOXIN LABORATORY OPERATIONS MANAGER
TRISH PARSLY
M HITCHCOCK, CLERK
D LAMBE, PURCH
S GREENDINE, RECPTNST
(C BREWER, M&S COORD)
GC/MS
S MILLS, GCMS MGR
M MEEHAN, TEAM LDR
S LOWRY, CHEMIST
M SKERTICH, CHEMIST
D THAL, CHEMIST
SAMPLE PREP
D McNELL, PREP GROUP LDR
L LOWER, CHEMIST
K WILSON, CHEMIST
B DEMERON, TECH
ADMIN SUPPORT
CHARLOTTE ROBBINS
QC/DOCUMENT CONTROL
L RAWLINS, ORL QCC
K JONES, ORL RECORDS MGT
C RIGELL, MBP QCC
J NORDQUIST, MBP QCC
M TYLER, SAL QCC
PROJECT MANAGEMENT
SHEREE' SCHNEIDER
S ADERHOLDT, MBP PC
A JONES, PA (T)
V GRAVES, ORL PC
C JACKSON, PA
J LANDSHOF, MBP PC
S ROBBINS, PA
B CURRY, MBP PM
M CARABELLAS, PC
C BAGGETT, ORL PC
J REYNOLDS, PA
J VANCE, SAL PC
M CONRAD, PA
SAMPLE CONTROL
A CHARLES, GROUP LDR-MBP
K KLEIN, CHEMIST
D BLOOMQUIST, TECH (T)
A BLANFORD, TECH (T)
D ALLEN, TECH
J VANDERMOORE, TEAM LDR-ORL
M MAYHEW, TECH
R FORD, MP TECH
REPORT PRODUCTION
C CONNER, TEAM LDR-ORL
B VITATOE, CLERK
K SIDES, CLERK
(VACANT), TEAM LDR-MBP
D COWARD, CLERK
J PATTERSON, CLERK
J KIRBY, CLERK
S MELAS, CLERK
S MT, CLERK (T)
SYSTEMS MANAGEMENT
BRUCE WAGNER
B ROBINSON, MBP PROGRM
J LONGMIRE, ORL PROGRM
T McELROY, MBP PROGRM
D HULL, ORL PROGRM
R McGILL, SUPPORT
M SEABOLT, TECH DIR
ACCOUNTING MANAGER
DARRYL PERKINS
B NICHOLS, ACCT
RADIOLOGICAL OPERATIONS MANAGER
TODD HARDT
P JACKSON, RECPTNST
D KING, SECRETARY
J CRAFT, PURCH
RADIOCHEM PRODUCTN
R HULL, GROUP LDR
CARRIER GROUP
R TERRY, TEAM LDR-CARRIER
L MEISCHEER, CHEMIST
S BILLINGS, CHEMIST
T HIE, CHEMIST
R FOLEY, TECH
B SHAW, CHEMIST
S FOUST, TECH (T)
S HARPER, TECH
TRACER GROUP
B CRUM, TEAM LDR-TRACER
A GLENN, CHEMIST
T CLARK, CHEMIST
L GREEN, CHEMIST
A TURNER, CHEMIST
P DAVIS, TECH
S LANGHLIN, TECH
K THOMAS, TECH
CHEMICAL OPERATIONS MANAGER
CHARLES RUSSELL
K ECCLES, PURCH
(VACANT), RECPTNST
GC/MS
S MILLER, GROUP LDR
R LaFORD, CHEMIST
V HOOD, CHEMIST
P LAWSON, CHEMIST
M POTOCEK, CHEMIST
R ROBERTS, CHEMIST
D CODY, CHEMIST
B CORBIN, CHEMIST
R JOSE, RECORDS MGT
P STOCKTON, RECORDS MGT
ORG PREP/CONN C/HPLC
J LAUHOFF, GROUP LDR
ORGANIC PREP
M HAYDEN, TEAM LDR-A
M RAMSEY, CHEMIST
K FOX, CHEMIST
L JONES, CHEMIST
L McMEATER, CHEMIST
L WHEELER, TEAM LDR-B
K BELLAMY, CHEMIST
M PATTY, TECH (T)
A MILLER, TECH (T)
CONN CHEM/HPLC
J KLENS, TEAM LDR (HPLC)
B COCHRAN, CHEMIST (HPLC)
J RYAN, CHEMIST (HPLC)
A RIBET, CHEMIST
R McDONALD, TECH
J MULLINS, TECH
D WHEELER, CHEMIST
B LANDSHOF, TECH (T)
GC
D LONGNECKER, GROUP LDR
S GOSS, TEAM LDR-GC VOA
S HARRIS, CHEMIST
J JONES, CHEMIST
B WILES, CHEMIST (T)
K HENRY, CHEMIST
C CRUM, TEAM LDR-CLP GC
D GATES, CHEMIST
C HYNATT, CHEMIST
S WANI, CHEMIST (T)
R JOHNSON, CHEMIST
R HENRY, CHEMIST
K BULL, CHEMIST (ASC)
R MILLER, TEAM LDR-GC EXT
J JONES, CHEMIST
L LIU, CHEMIST
METALS
S CHUNG, GROUP LDR
M DURHAM, CHEMIST (GF)
L McMAHAN, CHEMIST (ICP)
S ROSENBERG, CHEMIST (WARD)
K SZELAGOWSKI, CHEMIST (PREP/CV)
C KAIKER, CHEMIST (ICP/CV)
J ARMSTRONG, CHEMIST (GF)
D WILBURN, CHEMIST (WARD)
F FORAUZON, CHEMIST (PREP)
K HOLLOWAY, CHEMIST (ICP)
C YANDELL, CHEMIST (WARD/GF)
J JOHNSON, TECH (T)(GF)
V STEPP, CHEMIST (ASC)

APPENDIX

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INSTRUMENT LIST

TABLE 2-1
INSTRUMENT INVENTORY
MIDDLEBROOK LABORATORY

INSTRUMENT TYPE	MANUFACTURER	MODEL	AUTOSAMPLER	AGE
GC/MS	Finnigan	4500	HP 7673	7
	Finnigan	Incos 500	Tekmar ALS2016	2
	Finnigan	Incos XL	CTC Anal. A2005	1
	VG	Trio I	HP 7673A	3
	Hewlett Packard	5970	Tekmar ALS2016	10
	Hewlett Packard	5970	Tekmar ALS2016	10
GC	Hewlett Packard	5890 ECD K/L	HP 7673A	1
	Hewlett Packard	5890 NPD	HP 7673	2
	Hewlett Packard	5890 FID	HP 7673	3
	Hewlett Packard	5890 FID	HP 7673	3
	Hewlett Packard	5890 ECD M/N	HP 7673A	6
	Hewlett Packard	5890 ECD C/D	HP 7673	3
	Hewlett Packard	5890 ECD G/H	HP 7673	3
	Varian	3740 ECD A/B	Series 8000	7
	Varian	3600 PID&HALL	Tekmar ALS2016	4
	Varian	3400 PID&HALL	Tekmar ALS2016	3
	Varian	3400 FID & PID	Tekmar ALS2016	2
	Varian	3700 FID & PID	Tekmar ALS2016	8
	Varian	3700 ECD E/F	Series 8000	10
	Varian	3700 ECD 5/6	Series 8000	8

TABLE 2-1
INSTRUMENT INVENTORY
MIDDLEBROOK LABORATORY
(continued)

INSTRUMENT TYPE	MANUFACTURER	MODEL	AUTOSAMPLER	AGE
GC (continued)	Varian	3700 ECD 1/2	Series 8000	10
	Varian	3400 PID&HALL	Tekmar ALS2016	3
METALS	Perkin Elmer	Zeeman 5100-A	PE AS-60	3
	Perkin Elmer	Zeeman 5100-B	PE AS-60	2
	Perkin Elmer	PC 5100-C	PE AS-60	3
	Thermo Jarrell Ash	1100		5
	Thermo Jarrell Ash	ICAP 61E		2
HPLC	Perkin Elmer	LC-235	PE LC-600	7
	Perkin Elmer	LC-235	PE LC-600	7
	Perkin Elmer	LC-95	PE ISS-100	10
	Waters	200	WISP 712	1
WW	Coleman	Model 35		3.5
	Dormann	Xertex		
	Hach	DR2		6
	Hach	2100A		6
	Lachat	1300-000	Lachat 1100-000	
	Lachat	1300-000	Lachat 1100-000	
	Lachat	Quick Chem AE	Lachat 2100	1
	OI Corporation	700		7
	Orion	SA520		6

TABLE 2-1
INSTRUMENT INVENTORY
MIDDLEBROOK LABORATORY
(continued)

INSTRUMENT TYPE	MANUFACTURER	MODEL	AUTOSAMPLER	AGE
	Orion	920A		
WW (Continued)	YSI	Model 35	Lachat 2100	4
Organic Prep	ABC 1	GPC Autoprep Model 1002B		3
	ABC 2	GPC Autoprep Model 1002B		3
	ABC A	GPC Autoprep Model 1002B		3
	ABC B	GPC Autoprep Model 1002B		1
	Hewlett Packard	5890 Series II	HP 7673	2
	Zymark	Turbo VAP II 2		1
	Zymark	Turbo VAP II 1		4
	Zymark	Turbo VAP 7		4
	Zymark	Turbo VAP 6		4
	Zymark	Turbo VAP 5		4
	Zymark	Turbo VAP 4		4
	Zymark	Turbo VAP 3		4
	Zymark	Turbo VAP 2		4
	Zymark	Bench. Work.		1
	Zymark	Turbo VAP 1		4

TABLE 2-1
INSTRUMENT INVENTORY
MIDDLEBROOK LABORATORY
(continued)

INSTRUMENT TYPE	MANUFACTURER	MODEL	AUTOSAMPLER	AGE
	Tekmar	Sonicator TM600-2		
	Tekmar	Sonicator TM600-2		
Organic Prep (Continued)	Tekmar	Sonicator TM500		
	Tekmar	Sonicator TM500		

APPENDIX

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STANDARD OPERATING PROCEDURE LIST

ITAS-KNOXVILLE LABORATORY

STANDARD OPERATING PROCEDURES INDEX

SOP NO.	REV NO.	REV. DATE	TITLE
AD910306R0	0	3/7/91	Transfer of Document Files to Off-Site Storage
AD910312R1	1	11/1/91	General Health and Safety Practices for Tasks Performed in the ITAS-Knoxville Laboratory
AD910313R1	1	5/28/91	Internal Chain-of-Custody and Sample Security for ITAS-Knoxville
CD900420R1	1	5/14/93	NJDEPE X-26174 IFB Specific Requirements for Sample Container Preparation and Shipping, Sample Receipt and Logging, Internal Chain of Custody and Sample Storage
iC841213R3	3	5/10/93	GC Sample Analysis and Tracking
iC860529R3	3	12/30/91	GC Data Review for CLP Data Package - SOW OLMO1
T-CO-0020	1	5/14/93	Extraction of Low Level Semivolatiles from Soil/Sediment Samples Using CLP Protocol
T-CO-0021	1	5/10/93	Extraction of Medium Level Semivolatile Compounds from Soil/Sediment Samples Using CLP Protocol
T-CO-0024	1	5/14/93	Semivolatile Extractions by Continuous Liquid-Liquid Extraction, CLP Protocol
T-CO-0030	0	5/10/93	Analysis of Pesticides and PCBs by the CLP Protocol
T-CO-3640	0	5/10/93	Gel Permeation Chromatography Organic Extract Cleanup Procedure - Semivolatiles
TAS-KN-GC-6407	1	9/1/93	Gas Chromatography Analysis of Pesticides and PCBs by Contract Laboratory Protocol SOW 3/90
TAS-KN-MS-6821	1	9/1/93	GC/MS Semivolatile Organic Analysis by USEPA SW-846 Method 8270
TAS-KN-MS-6823	1	9/1/93	GC/MS Semivolatile Organic Analysis by Contract Laboratory Protocol OLMO1.0, SOW 3/90
AS-KN-MS-6831	1	9/2/93	GC/MS Volatile Organic Analysis by USEPA SW-846 Method 8240
AS-KN-MS-6834	1	9/1/93	GC/MS Volatile Organic Analysis by Contract Laboratory Protocol OLMO1.0, SOW 3/90

ITAS-KNOXVILLE LABORATORY

STANDARD OPERATING PROCEDURES INDEX

SOP NO.	REV NO.	REV. DATE	TITLE
S-KN-MT-6200	1	9/1/93	ICP Method for Trace Element Analysis of Water and Wastes
S-KN-MT-6201	1	9/1/93	Analysis of Trace Metals by Furnace Atomic Absorption Spectrometry by CLP
S-KN-MT-6202	1	9/1/93	Flame Atomic Absorption Method for Potassium Analysis by CLP SOW 3/90
S-KN-MT-6203	1	9/1/93	Analysis of Mercury in Liquid Waste by CLP SOW 3/90 and SW-846
S-KN-MT-6211	1	9/1/93	Sample Preparation for Metals Analysis by CLP SOW 3/90
S-KN-OP-6690	1	9/1/93	Toxicity Characteristic Leaching Procedure for Metals, Semivolatiles, Pesticides, PCBs and Herbicides
S-KN-WC-6000	1	8/31/93	Alkalinity Analyses: Total, Bicarbonate, Carbonate and Hydroxide Alkalinity
S-KN-WC-6001	1	8/31/93	Filterable Residue Analysis (Gravimetric, Dried at 180°C)
S-KN-WC-6002	1	8/31/93	Nonfilterable Residue Analysis (Gravimetric, Dried at 103-105°C
S-KN-WC-6003	1	8/31/93	Analysis of Total Cyanide in Water, Soil and Sediment Media by USEPA CLP Protocol (Automated)
S-KN-WC-6004	1	8/31/93	Chloride by Flow Injection Analysis
S-KN-WC-6005	1	8/31/93	Sulfate by Flow Injection Analysis (Automated, Methylthymol Blue, AAIII
S-KN-WC-6006	1	8/31/93	Total and Ortho Phosphorus by Flow Injection Analysis
S-KN-WC-6007	1	9/1/93	Nitrate/Nitrite by Flow Injection Analysis (Automated, Cadmium Reduction)
S-KN-WC-6008	1	9/1/93	Analysis of Total Organic Carbon in Water (UV Promoted, Persulfate Oxidation)
S-KN-WC-6009	1	9/1/93	Analysis of Oil and Grease and Total Petroleum Hydrocarbons (TPH, Gravimetric)

ITAS-KNOXVILLE LABORATORY

STANDARD OPERATING PROCEDURES INDEX

SOP NO.	REV NO.	REV. DATE	TITLE
ITAS-KN-WC-6010	1	9/1/93	Ammonia Nitrogen Analysis (Nesslerization)
ITAS-KN-WC-6011	1	9/1/93	Determination of Total Organic Nitrogen (TON)
ITAS-KN-WC-6012	1	9/1/93	Analysis of Ph in Water and Soil Samples
TAS-KN-WC-6014	1	9/1/93	Total Kjeldahl Nitrogen (TKN) by Nesslerization
TAS-KN-WC-6016	1	9/1/93	Fluoride Analysis by Ion Selective Electrode (ISE)
TAS-KN-WC-6017	1	9/1/93	Analysis of Sulfides in Waters (Titrimetric)
TAS-KN-WC-6020	1	9/1/93	Analysis of Total Organic Halides (TOX)
TAS-KN-WC-6022	1	9/1/93	Analysis of Sulfite in Waters
TAS-KN-WC-6023	1	9/1/93	Total Residue
TAS-KN-WC-6030	1	9/1/93	Hexavalent Chromium in Water and Soil
TAS-KN-WC-6031	1	9/1/93	Analysis of Phenolic Compounds in Water and Soil (Automated)
TAS-KN-WC-6038	1	9/1/93	Conductivity
TAS-KN-WC-6039	1	9/1/93	Residual Chlorine
TAS-KN-WC-6040	1	9/1/93	Ignitability by Penske-Martens Closed Flash Tester
TAS-KN-WC-6041	1	9/1/93	Ignitability of Liquids by Setaflash
TAS-KN-WC-6053	1	9/1/93	Hardness by Titration
TMS390V	0	6/15/92	GC/MS Volatile Organic Analysis by EPA CLP 3/90 SOW W/Revisions

ITAS-KNOXVILLE LABORATORY

STANDARD OPERATING PROCEDURES INDEX

SOP NO.	REV NO.	REV. DATE	TITLE
KN-CO-0006R0	0	5/10/93	Gel Permeation Chromatography Organic Extract Cleanup Procedure - Pesticides/PCBs
KN-GC-0003R0	0	5/10/93	Standard Preparation Notebook Usage - GC
KN-HS-0012R1	1	1/10/92	Hazardous Waste Management Policy
KN-HS-0013R0	0	1/10/92	Waste Characterization and Categorization
KN-HS-0014R0	0	1/10/92	Waste Accumulation
KN-HS-0015R0	0	1/10/92	Waste Packaging and Storage
KN-HS-0016R0	1	1/10/92	Waste Shipping and Manifesting
KN-HS-0017R0	1	1/10/92	Waste Minimization Policy
KN-MS-0001R0	0	5/10/93	Data Management and Handling in GC/MS and GC (Organics) Departments Under the USEPA CLP OLMO1
KN-OP-0001R0	0	5/10/93	Records Management - Organic Extractions
KN-OP-0002R0	0	5/10/93	Reagent Purity Check Procedures
KN-OP-0003R0	0	2/4/93	Sonicator Horn Tuning in Organic Extractions
KN-OP-0004R0	0	5/10/93	Sample Handling Protocol - Organic Extractions
KN-QC-0001R0	0	2/19/93	Pipettor Calibration - Gravimetric Method
KN-QC-0002R0	0	5/10/93	Personnel Training and Qualifications
KN-QC-0003R0	0	5/10/93	Facility Security
KN-QC-0004R0	0	2/1/93	Data Recording Practices

ITAS-KNOXVILLE LABORATORY

STANDARD OPERATING PROCEDURES INDEX

SOP NO.	REV NO.	REV. DATE	TITLE
KN-QC-0005R0	0	5/10/93	Nonconformance and Corrective Action
KN-QC-0006R2	0	5/10/93	Balance Calibration and Use of Class S Weights
KN-QC-0008R0	0	5/10/93	Quality and Operations Records Retention
KN-QC-0009R0	0	5/10/93	Calibration of Thermometers
KN-QC-0010R0	0	5/10/93	Temperature Monitoring
KN-QC-0015R0	0	5/10/93	Determination of MDL, CRDL and CRQL
KN-SC-0001R0	0	5/10/93	Receipt and Logging In of USEPA CLP and SAS Samples
KN-SC-0003R0	0	5/10/93	Procedure and Documentation Requirements for Sample Archiving and Disposal
M_841218R2	2	5/10/93	GC/MS Sample Tracking
M_870715R1	0	5/10/93	Records Management - GC/MS
M_880520R2	2	9/3/91	EPA-CLP and SAS Case File Assembly
M_880630R2	2	9/3/91	Shipment of EPA CLP and SAS Deliverable Packages
M_911220R0	0	12/20/91	Review of EPA-CLP OLM01 Diskette Deliverables
OP841214R3	3	12/11/91	Glassware Cleanup for Organic Extractions
OP910904R1	0	5/10/93	Extraction Procedures for USEPA Target Compound List Chlorinated Pesticides and Aroclors by USEPA CLP 3/90 SOW Protocol
OP910925R1	0	5/10/93	Extraction of Pesticides/PCBs from Soil/Sediment Samples Using EPA CLP SOW OLM01
OP911230R0	0	12/30/91	GC FID Screening

ITAS-KNOXVILLE LABORATORY

STANDARD OPERATING PROCEDURES INDEX

SOP NO.	REV NO.	REV. DATE	TITLE
841113R2	2	9/5/91	Sample Storage for EPA Contract Laboratory Program
841214R1-6	1	2/13/87	Water Purification System Monitoring

APPENDIX

KNOXVILLE - 4

ANALYTICAL METHODS

TABLE KN-4-1
KNOXVILLE LABORATORY
Wet Chemistry Methods

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Acidity	Water	Method 305.2	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Alkalinity	Water	Method 310.1	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Ammonia	Water	Method 350.2	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Method 350.2 [1]
Biochemical Oxygen Demand (BOD)	Water	Method 405.1	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Bromide	Water	Method 300.0	Not Applicable	Not Applicable	Not Applicable

TABLE KN-4-1
KNOXVILLE LABORATORY
Wet Chemistry Methods
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Bromide (continued)	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Method 300.0 [2]
Chemical Oxygen Demand (COD)	Water	Not Applicable	Not Applicable	Not Applicable	HACH
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Chloride	Water	Method 300.0 Method 325.3	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Method 300.0 [2] Method 325.3 [3]
Conductivity	Water	Method 120.1	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cyanide (Amenable)	Water	Method 335.1	Method 9010	Not Applicable	Not Applicable

TABLE KN-4-1
KNOXVILLE LABORATORY
Wet Chemistry Methods
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Cyanide (Amenable) (continued)	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cyanide (Free)	Water	Not Applicable	Not Applicable	Not Applicable	Standard Methods 412 H
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Standard Methods 412 H
Cyanide	Water	Method 335.2	Method 9010	Method ILM01.0	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 9010[4]	Method ILM01.0	Not Applicable
Dissolved Oxygen	Water	Method 360.1	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Dissolved Organic Carbon (DOC)	Water	Method 415.1	Method 9060	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE KN-4-1
KNOXVILLE LABORATORY
Wet Chemistry Methods
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Dissolved Organic Carbon (DOC) (continued)	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Fluoride	Water	Method 300.0 Method 340.2	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Method 300.0 [2] Method 340.2 [3]
Hardness	Water	Not Applicable	Not Applicable	Not Applicable	SM 314A
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Kjeldahl Nitrogen (total)	Water	Method 351.3	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Method 351.3[3]
Nitrite (NO_2)	Water	Method 300.0 (non-preserved) Method 353.2 (LACHAT) (preserved)	Not Applicable	Not Applicable	Not Applicable

TABLE KN-4-1
KNOXVILLE LABORATORY
Wet Chemistry Methods
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Nitrite (NO_2) (continued)	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Nitrate (NO_3)	Water	Method 300.0 (non-preserved) Method 353.2 (LACHAT) (preserved)	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Nitrate plus Nitrite	Water	Method 353.2 (LACHAT)	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Method 353.2 [3] (LACHAT)
Oil and Grease	Water	Method 413.1	Method 9070	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Method 9071	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 9071	Not Applicable	Not Applicable

TABLE KN-4-1
KNOXVILLE LABORATORY
Wet Chemistry Methods
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Orthophosphate	Water	Method 300.0 Method 365.3 (LACHAT)	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Method 365.3 [3] (LACHAT)
pH	Water	Method 150.1	Method 9040	Method OLM01.8	Not Applicable
	Liquid	Not Applicable	Method 9040	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 9040	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Method 9045	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 9045	Method OLM01.8	Not Applicable
Phenol	Water	Method 420.2 (LACHAT)	Method 9065 (LACHAT)	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 9065[4] (LACHAT)	Not Applicable	Not Applicable
Phosphorus (Total)	Water	Method 365.3	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge,Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE KN-4-1
KNOXVILLE LABORATORY
Wet Chemistry Methods
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Residual Chlorine	Water	Not Applicable	Not Applicable	Not Applicable	HACH
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Sulfate (SO_4)	Water	Method 300.0 Method 375.4	Method 9038	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Sulfide (SO)	Water	Method 376.1	Method 9030	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Method 376.1 [6]
Sulfite (SO_3)	Water	Method 377.1	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Method 377.1 [6]
Total Organic Carbon (TOC)	Water	Method 415.1	Method 9060	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE KN-4-1
KNOXVILLE LABORATORY
Wet Chemistry Methods
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
TOC (continued)	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 9060 (7)	Not Applicable	Not Applicable
Total Dissolved Solids	Water	Method 160.1	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Total Settleable Solids	Water	Method 160.5	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Total Solids	Water	Method 160.3	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Total Suspended Solids	Water	Method 160.2	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE KN-4-1
KNOXVILLE LABORATORY
Wet Chemistry Methods
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Suspended Solids (continued)	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Total Volatile Solids	Water	Method 160.4	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Turbidity	Water	Method 180.1	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE KN-4-1
KNOXVILLE LABORATORY
Wet Chemistry Methods
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Turbidity (continued)	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable

(1) 5 g soil is added to 250 ml DI water in the distillation flask; the distillation and analysis procedure is followed as described in the reference EPA method(s).

(2) Soil is leached.

(3) 1 g soil is added to 100 ml DI water and sonicated for 30 minutes in a water bath. A portion of the leachate is then decanted, filtered if necessary, and analyzed according to the referenced method(s). The results are reported as water leachable concentrations of the analytes.

(4) 5 g soil is added to 500 ml DI water in the distillation flask; the distillation and analysis procedure is then followed as described in the referenced EPA method(s).

(5) 1 g soil is added to 50 ml DI water in the digestion apparatus: the digestion, distillation, and analysis procedure is then followed as described in the referenced EPA method.

(6) 2 g soil is added to an appropriate volume of DI water (200 ml for sulfide analysis, 50 ml for sulfite analysis); the solution is agitated with a magnetic stirrer and titrated in accordance with the referenced EPA methods.

(7) Samples are weighed in a glass ampule and mixed with a phosphoric and sodium persulfate reagent solution. The phosphoric acid immediately converts all inorganic carbon to carbon dioxide (CO_2) which is purged from the ampule with oxygen. The ampule is sealed under oxygen and heated to approximately 100° in a water bath for 30 minutes. The heating process converts the organic carbon to CO_2 which is trapped in the sealed ampule. The samples are analyzed according to the referenced EPA methods by an O. I. Corporation Total Organic Carbon Analyzer, Model 700. The carbon is quantified by an infrared detector in the analyzer by measuring the absorbance of CO_2.

TABLE KN-4-2
KNOXVILLE LABORATORY
Metals Sample Preparation Methods

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Toxicity Characteristic Leaching Procedure (TCLP)	Water	Not Applicable	Method 1311	Not Applicable	Not Applicable
	Liquid	Not Applicable	Method 1311	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 1311	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Method 1311	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 1311	Not Applicable	Not Applicable
ICAP Metals	Water	Not Applicable	Method 3005 Method 3010	Method ILMO1.0	Not Applicable
	Liquid	Not Applicable	Method 3040	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 3010	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Method 3050	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 3050	Method ILMO1.0	Not Applicable
CVAA Mercury	Water	Method 245.1	Method 7470	Method ILMO1.0	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Extract	Not Applicable	Method 7470	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment	Not Applicable	Method 7471	Not Applicable	Not Applicable
	Soil	Method 245.5	Method 7471	Method ILMO1.0	Not Applicable

TABLE KN-4-2
KNOXVILLE LABORATORY
Metals Sample Preparation Methods

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
GFAA Metals	Water	Element / Method Arsenic 206.2 Antimony 204.2 Cadmium 213.2 Chromium 218.2 Lead 239.2 Nickel 249.2 Selenium 270.2 Silver 272.2 Thallium 279.2	Method 3020	Method ILMO1.0	Not Applicable
	Liquid	Not Applicable	Method 3020	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 3020	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solid, Sediment		Method 3050	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 3050	Method ILMO1.0	Not Applicable

TABLE KN-4-3
KNOXVILLE LABORATORY
Inductively Coupled Plasma Emission Spectroscopy Methods

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Al, Sb, As, Ba, Be, Bo, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, Os, K, Se Si, Ag, Na, Sr, Tl, Sn, Ti, V, Zn	Water	Not Applicable	Method 6010	Method ILM01.0	Not Applicable
	Liquid	Not Applicable	Method 6010	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 6010	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 6010	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 6010	Method ILM01.0	Not Applicable

TABLE KN-4-4
KNOXVILLE LABORATORY
Graphite Furnace Atomic Absorption Methods

Parameter	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Antimony	Water	Method 204.2	Method 7041	Method ILMO1.0	Not Applicable
	Liquid	Not Applicable	Method 7041	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 7041	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 7041	Method ILMO1.0	Not Applicable
Arsenic	Water	Method 206.2	Method 7060	Method ILMO1.0	Not Applicable
	Liquid	Not Applicable	Method 7060	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 7060	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 7060	Method ILMO1.0	Not Applicable
Cadmium	Water	Method 213.2	Method 7131	Not Applicable	Not Applicable
	Liquid	Not Applicable	Method 7131	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 7131	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 7131	Not Applicable	Not Applicable
Chromium	Water	Method 218.2	Method 7191	Not Applicable	Not Applicable

TABLE KN-4-4
KNOXVILLE LABORATORY
Graphite Furnace Atomic Absorption Methods

Parameter	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Chromium (continued)	Liquid	Not Applicable	Method 7191	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 7191	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 7191	Not Applicable	Not Applicable
Lead	Water	Method 239.2	Method 7421	Method ILMO1.0	Not Applicable
	Liquid	Not Applicable	Method 7421	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 7421	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 7421	Method ILMO1.0	Not Applicable
Nickel	Water	Method 249.2	Not Applicable	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge Solids, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Selenium	Water	Method 270.2	Method 7740	Method ILMO1.0	Not Applicable
	Liquid	Not Applicable	Method 7740	Method ILMO1.0	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE KN-4-4
KNOXVILLE LABORATORY
Graphite Furnace Atomic Absorption Methods

Parameter	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Selenium (continued)	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 7740	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 7740	Method ILMO1.0	Not Applicable
Silver	Water	Method 272.2	Method 7761	Not Applicable	Not Applicable
	Liquid	Not Applicable	Method 7761	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 7761	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 7761	Not Applicable	Not Applicable
Thallium	Water	Method 279.2	Method 7841	Method ILMO1.0	Not Applicable
	Liquid	Not Applicable	Method 7841	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 7841	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 7841	Method ILMO1.0	Not Applicable

TABLE KN-4-5
KNOXVILLE LABORATORY
Cold Vapor Atomic Absorption Methods

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Mercury	Water	Method 245.1	Method 7470	Method ILM01.0	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Extract	Not Applicable	Method 7470	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 7471	Not Applicable	Not Applicable
	Soil	Method 245.5	Method 7471	Method ILM01.0	Not Applicable

TABLE KN-4-6
KNOXVILLE LABORATORY
Organic Sample Preparation Methods

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Volatiles by GC/MS	Water	Method 624	Method 5030	Method OLMO1.8	Not Applicable
	Liquid	Not Applicable	Method 5030	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 5030	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 5030	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 5030	Method OLMO1.8	Not Applicable
Volatiles by GC	Water	Method 601	Method 5030	Not Applicable	Not Applicable
	Liquid	Not Applicable	Method 5030	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 5030	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 5030	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 5030	Not Applicable	Not Applicable
Aromatic Volatiles by GC	Water	Method 602	Method 5030	Not Applicable	Not Applicable
	Liquid	Not Applicable	Method 5030	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 5030	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 5030	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 5030	Not Applicable	Not Applicable
Semivolatiles by GC/MS	Water	Method 625	Method 3510 Method 3520	Method OLM01.8	Not Applicable
	Liquid	Not Applicable	Method 3580	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 3510 Method 3520	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 3540 Method 3550	Not Applicable	Not Applicable

TABLE KN-4-6
KNOXVILLE LABORATORY
Organic Sample Preparation Methods
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Semivolatiles by GC/MS (continued)	Soil	Not Applicable	Method 3540 Method 3550	Method OLMO1.8	Not Applicable
Pesticides/ PCBs by GC	Water	Method 608	Method 3510 Method 3520	Method OLMO1.8	Not Applicable
	Liquid	Not Applicable	Method 3580	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 3510 Method 3520	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 3540 Method 3550	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 3540 Method 3550	Method OLMO1.8	Not Applicable
Herbicides by GC	Water	Not Applicable	Method 8150	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 8150	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Low Boiling Petroleum Hydrocarbons by GC	Water	Not Applicable	Method 5030	Not Applicable	TN GRO Method
	Liquid	Not Applicable	Method 5030	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 5030	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 5030	Not Applicable	TN GRO Method
High Boiling Petroleum Hydrocarbons by GC	Water	Not Applicable	Method 3510 Method 3520	Not Applicable	TN DRO Method
	Liquid	Not Applicable	Method 3580	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 3540 Method 3550	Not Applicable	Not Applicable

TABLE KN-4-6
KNOXVILLE LABORATORY
Organic Sample Preparation Methods
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
High Boiling Petroleum Hydrocarbons by GC (continued)	Soil	Not Applicable	Method 3540 Method 3550	Not Applicable	TN DRO Method
Nitroaromatics by HPLC	Water	Not Applicable	Method 8330	Not Applicable	USATHAMA UW-11
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediments	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 8330	Not Applicable	USATHAMA LW-06
Polynuclear Aromatic Hydrocarbons by HPLC	Water	Method 610	Method 3510 Method 3520	Not Applicable	Not Applicable
	Liquid	Not Applicable	Method 3580	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 3540 Method 3550	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 3540 Method 3550	Not Applicable	Not Applicable
Nitroaromatics by GC	Water	Method 609	Method 3510 Method 3520	Not Applicable	Not Applicable
	Liquid	Not Applicable	Method 3580	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 3540 Method 3550	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 3540 Method 3550	Not Applicable	Not Applicable
Organophosphorus Pesticides by GC	Water	Not Applicable	Method 3510 Method 3520	Not Applicable	Not Applicable
	Liquid	Not Applicable	Method 3580	Not Applicable	Not Applicable

TABLE KN-4-6
KNOXVILLE LABORATORY
Organic Sample Preparation Methods
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Organophosphorus Pesticides by GC (continued)	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 3540 Method 3550	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 3540 Method 3550	Not Applicable	Not Applicable

TABLE KN-4-7
KNOXVILLE LABORATORY
Organic Methods

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Volatiles by GC/MS	Water	Method 624	Method 8240 & 8260	Method OLMO1.8 & OLV01.0	Method 524.2
	Liquid	Not Applicable	Method 8240	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 8240	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 8240	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 8240 & 8260	Method OLM01.8	Not Applicable
Halogenated Volatiles by GC	Water	Method 601	Method 8010	Not Applicable	Not Applicable
	Liquid	Not Applicable	Method 8010	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 8010	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 8010	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 8010	Not Applicable	Not Applicable
Aromatic Volatiles by GC	Water	Method 602	Method 8020	Not Applicable	Not Applicable
	Liquid	Not Applicable	Method 8020	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 8020	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 8020	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 8020	Not Applicable	Not Applicable
Semivolatiles by GC/MS	Water	Method 625	Method 8270	Method OLM01.8	Not Applicable
	Liquid	Not Applicable	Method 8270	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 8270	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 8270	Not Applicable	Not Applicable

TABLE KN-4-7
KNOXVILLE LABORATORY
Organic Methods
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Semivolatiles by GC/MS (continued)	Soil	Not Applicable	Method 8270	Method OLM01.8	Not Applicable
Pesticides/ PCBs by GC	Water	Method 608	Method 8080	Method OLM01.8	Not Applicable
	Liquid	Not Applicable	Method 8080	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 8080	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 8080	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 8080	Method OLM01.8	Not Applicable
Herbicides by GC	Water	Not Applicable	Method 8150	Not Applicable	Not Applicable
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Method 8150	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Low Boiling Petroleum Hydrocarbons by GC	Water	Not Applicable	CA MOD. Method 8015	Not Applicable	TN GRO Method
	Liquid	Not Applicable	CA MOD. Method 8015	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids Sediment	Not Applicable	CA MOD. Method 8015	Not Applicable	Not Applicable
	Soil	Not Applicable	CA MOD. Method 8015	Not Applicable	TN GRO Method
High Boiling Petroleum Hydrocarbons by GC	Water	Not Applicable	CA MOD. Method 8015	Not Applicable	TN DRO Method
	Liquid	Not Applicable	CA MOD. Method 8015	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE KN-4-7
KNOXVILLE LABORATORY
Organic Methods
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
High Boiling Petroleum Hydrocarbons by GC (continued)	Domestic Waste, Industrial Waste Sludge, Solids, Sediment	Not Applicable	CA MOD. Method 8015	Not Applicable	Not Applicable
	Soil	Not Applicable	CA MOD. Method 8015	Not Applicable	TN DRO Method
Nitroaromatics by HPLC	Water	Not Applicable	Method 8330	Not Applicable	USATHAMA UW-11
	Liquid	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 8330	Not Applicable	USATHAMA LW-06
Polynuclear Aromatic Hydrocarbons by HPLC	Water	Method 610	Method 8310	Not Applicable	Not Applicable
	Liquid	Not Applicable	Method 8310	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 8310	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 8310	Not Applicable	Not Applicable
Nitroaromatics by GC	Water	Method 609	Method 8090	Not Applicable	Not Applicable
	Liquid	Not Applicable	Method 8090	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable
	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 8090	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 8090	Not Applicable	Not Applicable
Organophosphorus Pesticides by GC	Water	Not Applicable	Method 8141	Not Applicable	Not Applicable
	Liquid	Not Applicable	Method 8141	Not Applicable	Not Applicable
	TCLP Leachate	Not Applicable	Not Applicable	Not Applicable	Not Applicable

TABLE KN-4-7
KNOXVILLE LABORATORY
Organic Methods
(continued)

Analytical Parameters	Matrix	Methods			
		NPDES	RCRA (SW846)	CLP	Other
Organophosphorus Pesticides by GC (continued)	Domestic Waste, Industrial Waste, Sludge, Solids, Sediment	Not Applicable	Method 8141	Not Applicable	Not Applicable
	Soil	Not Applicable	Method 8141	Not Applicable	Not Applicable

APPENDIX

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PRACTICAL QUANTITATION LIMITS

TABLE KN-5-1
KNOXVILLE LABORATORY
Practical Quantitation Limits for
Wet Chemistry

Parameter	Method	Water PQL (mg/l except as noted)	Soil PQL (mg/kg)
Acidity	305.2	1	Not Applicable
Alkalinity	310.1	1	Not Applicable
Ammonia	350.2	0.1	5
BOD	405.1	1	Not Applicable
COD	HACH	1	Not Applicable
Bromide	300.0	0.4	0.4
Chloride	325.3 300.0	0.5 0.4	5.0 0.4
Chlorine, Total Residual	HACH	0.1	Not Applicable
Conductivity	120.1	1 umhos/cm	Not Applicable
Cyanide	335.2/9010/ILMO1.0	0.01	1
Cyanide, amenable	335.1/9010	0.01	Not Applicable
Dissolved Oxygen	360.1	1	Not Applicable
Fluoride	340.2 300.0	0.1 0.4	10 0.4
Hardness	SM 314A	0.2	Not Applicable
Nitrate	353.2 300.0	0.02 0.4	Not Applicable 0.4
Nitrate/Nitrite	353.2	0.02	3
Nitrite	353.2 300.0	0.02 0.4	Not Applicable
TKN	351.3	0.1	5
Oil & Grease	413.1/9070/9071	1	50
Orthophosphate	365.3 300.0	0.01 1.0	1 1.0
pH	150.1/9040/9045	0.01 Standard Units	0.01 Standard Units
Phenols	420.2/9065	0.02	1
Sulfate	375.4/9038 300.0	10 1.5	Not Applicable 1.5
Sulfide	376.1/9030	0.2	20
Sulfite	377.1	0.5	20
Total Dissolved Solids	160.1	1	Not Applicable
Total Settleable Solids	160.5	0.1 ml/liter	Not Applicable

Parameter	Method	Water PQL (mg/l except as noted)	Soil PQL (mg/kg)
Acidity	305.2	1	Not Applicable
Total Solids	160.3	1	Not Applicable
Total Suspended Solids	160.2	1	Not Applicable
Total Volatile Solids	160.4	1	Not Applicable
TOC/DOC	415.1/9060	1	1
Turbidity	180.1	0.1 NTU	Not Applicable

TABLE KN-5-2
KNOXVILLE LABORATORY
ICAP Metals
Contract Required Detection Limits (CRDL)[1], Method Detection Limits (MDL)[2], and Practical Quantitation Limits (PQL)[3]

Element	CAS Number	Water CRDL (ug/L)	Soil CRDL[4] (mg/Kg)	Water MDL (ug/L)	Soil MDL[4] (mg/Kg)	Water PQL (ug/L)	Soil PQL[4] (mg/Kg)
Aluminum	7429-90-5	200	40	21		40	8
Antimony	7440-36-0	60	12	7.4		30	6
Arsenic	7440-38-2	10	2	18		30	6
Barium	7440-39-3	200	40	0.4		2	0.4
Beryllium	7440-41-7	5	1	0.2		1	0.2
Boron	7440-42-8			6.6		10	2
Cadmium	7440-43-9	5	1	1.5		5	1
Calcium	7440-70-2	5000	1000	19		30	6
Chromium	7440-47-3	10	2	3.6		10	2
Cobalt	7440-48-4	50	10	4.6		20	4
Copper	7440-50-8	25	5	2.6		10	2
Iron	7439-89-6	100	20	2.4		10	2
Lead	7439-92-1	3	0.6	12		30	6
Lithium	7439-93-2			4.0		5	20
Magnesium	7439-95-4	5000	1000	24		30	6
Manganese	7439-96-5	15	3	0.5		2	0.4
Molybdenum	7439-98-7			5.2		10	2
Nickel	7440-02-0	40	8	4.2		20	4
Potassium	7440-09-7	5000	1000	630		1000	200
Selenium	7782-49-2	5	1	40		60	12
Silicon	7440-21-3			15		20	2
Silver	7440-22-4	10	2	3.8		5	1
Sodium	7440-22-4	5000	1000	30		200	40
Strontium	7440-24-6			0.2		1	0.2
Thallium	7440-28-0	10	2	27		3	6
Tin	7440-31-5					20	4
Titanium	7440-32-6			1.5		3	0.6

TABLE KN-5-2
KNOXVILLE LABORATORY
ICAP Metals
Contract Required Detection Limits (CRDL)[1], Method Detection Limits (MDL)[2], and Practical Quantitation Limits (PQL)[3]

Element	CAS Number	Water CRDL (ug/L)	Soil CRDL[4] (mg/Kg)	Water MDL (ug/L)	Soil MDL[4] (mg/Kg)	Water PQL (ug/L)	Soil PQL[4] (mg/Kg)
Vanadium	7440-62-2	50	10	2.2		10	2
Zinc	7440-66-6	20	4	1.3		5	1

[1] CRDLs apply to analyses performed in accordance with the USEPA CLP Statement of Work for Inorganics Analysis, Document Number ILMO1.0

[2] The method detection limit study was performed on 12/02/92 using SW-846 method 6010.

[3] PQLs were estimated from the results of water MDL determinations.

[4] Soil detection limits are based on wet weight of sample and will be higher when converted to a dry weight basis.

TABLE KN-5-3
KNOXVILLE LABORATORY
AA Metals
Contract Required Detection Limits (CRDL)[1], Method Detection Limits (MDL)[2], and Practical Quantitation Limits (PQL)[3]

Element	CAS Number	Water CRDL (ug/L)	Soil CRDL[4] (mg/Kg)	Water MDL (ug/L)	Soil MDL[4] (mg/Kg)	Water PQL (ug/L)	Soil PQL[4] (mg/Kg)
Antimony	7440-36-0	60	12	4.5	0.36	6	0.6
Arsenic	7440-38-2	10	2	1.7	0.091	3	0.3
Cadmium	7440-43-9	5	1			0.2	0.6
Chromium	7440-47-3	10	2			3	0.6
Lead	7439-92-1	3	0.6	1.9	0.098	3	0.3
Nickel	7440-02-0	40	8			3	
Selenium	7782-49-2	5	1	0.50	0.023	3	0.3
Silver	7440-22-4	10	2	6.4	0.64	8	0.8
Thallium	7440-28-0	10	2	2.38	0.204	3	0.3
Mercury[5]	7439-97-6	0.2	0.1	0.175	0.013	0.2	0.1

[1] CRDLs apply to analyses performed in accordance with the USEPA CLP Statement of Work, Document Number ILM01.0.

[2] Method detection limit studies were performed during 10/92 - 11/92 using SW-846 7000 series methodology.

[3] PQLs were estimated from the results of water MDL determinations.

[4] Soil detection limits are based on wet weight of sample and will be higher when converted to a dry weight basis.

[5] Mercury is analyzed by cold vapor AA; all other elements are analyzed by graphite furnace AA.

TABLE KN-5-4
KNOXVILLE LABORATORY
Volatile Organics
Contract Required Detection Limits (CRDL)[1], Method Detection Limits (MDL)[2] and Practical Quantitation Limits (PQL)[3]

Analyte	CAS Number	Water CRDL (ug/L)	Soil CRDL[4] (ug/Kg)	Water MDL (ug/L)	Soil MDL[4] (ug/Kg)	Water PQL (ug/L)	Soil PQL[4] (ug/Kg)
Chloromethane	74-87-3	10	10	2.9		10	10
Bromomethane	74-83-9	10	10	2.6		10	10
Vinyl Chloride	75-01-4	10	10	3.1		10	10
Chloroethane	75-00-3	10	10	3.0		10	10
Methylene Chloride	75-09-2	10	10	3.1		5	5
Acetone	67-64-1	10	10	6.5		100	100
Carbon Disulfide	75-15-0	10	10	2.7		5	5
1,1-Dichloroethene	75-35-4	10	10	3.3		5	5
1,1-Dichloroethane	75-34-3	10	10	3.2		5	5
1,2-Dichloroethene (total)	540-59-0	10	10	3.4		5	5
Chloroform	67-66-3	10	10	3.1		5	5
1,2-Dichloroethane	107-06-2	10	10	3.4		5	5
2-Butanone	78-93-3	10	10	7.5		100	100
1,1,1-Trichloroethane	71-55-6	10	10	3.3		5	5
Carbon Tetrachloride	56-23-5	10	10	3.0		5	5
Vinyl Acetate		NA	NA	3.5		50	50
Bromodichloromethane	75-27-4	10	10	4.1		5	5
1,2-Dichloropropane	78-87-5	10	10	3.4		5	5
cis-1,3-Dichloropropene	10061-01-5	10	10	3.6		5	5
2-chloroethylvinylether	110-75-8	NA	NA	6.6		10	10
Trichloroethene	79-01-6	10	10	3.7		5	5
Dibromochloromethane	124-48-1	10	10	4.1		5	5
1,1,2-Trichloroethane	79-00-5	10	10	3.9		5	5
Benzene	71-43-2	10	10	3.0		5	5
trans-1,3-Dichloropropene	10061-02-6	10	10	5.7		5	5
Bromoform	75-25-2	10	10	3.7		5	5
4-Methyl-2-Pentanone	108-10-1	10	10	5.1		50	50

TABLE KN-5-4
KNOXVILLE LABORATORY
Volatile Organics
Contract Required Detection Limits (CRDL)[1], Method Detection Limits (MDL)[2] and Practical Quantitation Limits (PQL)[3]

Analyte	CAS Number	Water CRDL (ug/L)	Soil CRDL[4] (ug/Kg)	Water MDL (ug/L)	Soil MDL[4] (ug/Kg)	Water PQL (ug/L)	Soil PQL[4] (ug/Kg)
2-Hexanone	591-78-6	10	10	5.8		50	50
Tetrachloroethene	127-18-4	10	10	2.9		5	5
Toluene	108-88-3	10	10	3.6		5	5
1,1,2,2-Tetrachloroethane	79-34-5	10	10	3.6		5	5
Chlorobenzene	108-90-7	10	10	3.0		5	5
Ethylbenzene	100-41-4	10	10	3.0		5	5
Styrene	100-42-5	10	10	3.4		5	5
Xylene (total)	1330-20-7	10	10	3.2		5	5
Acrolein	107-02-8	10	10	7.9		10	10
Acrylonitrile	107-13-1	10	10	4.5		10	10
cis-1,2-Dichloroethene	156-59-2	10	10	2.8		10	10
Trichlorofluoromethane	75-69-4	10	10	3.5		10	10

[1] CRDLs apply to analyses performed in accordance with the USEPA CLP Statement of Work for Inorganics Analysis, Document Number OLMO1.8.

[2] The Method Detection Limit study for water was performed on 5/14/93 using SW-846 method 8240.

[3] Practical Quantitation Limits are taken from SW-846 Method 8240.

[4] Quantitation limits listed for soil are based on wet weight. The quantitation limits based on dry weight as required, will be higher.

TABLE KN-5-5
KNOXVILLE LABORATORY
Semivolatile Organics
Contract Required Detection Limits (CRDL)[1], Method Detection Limits (MDL)[2], and Practical Quantitation Limits (PQL)[3]

Analyte	CAS Number	Water CRDL (ug/L)	Soil CRDL[4] (ug/Kg)	Water MDL (ug/L)	Soil MDL[4] (ug/Kg)	Water PQL (ug/L)	Soil PQL[4] (ug/kg)
Phenol	108-95-2	10	330	0.9		10	330
bis(2-Chloroethyl)ether	111-44-4	10	330	1.1		10	330
2-Chlorophenol	95-57-8	10	330	1.8		10	330
1,3-Dichlorobenzene	541-73-1	10	330	1.9		10	330
1,4-Dichlorobenzene	106-46-7	10	330	2.1		10	330
Benzyl Alcohol	100-51-6	NA	NA	2.5		20	670
1,2-Dichlorobenzene	95-50-1	10	330	1.7		10	330
2-Methylphenol	95-48-7	10	330	1.7		10	330
bis(2-chloroisopropyl)ether	108-60-1	10	330	1.2		10	330
4-Methylphenol	106-44-5	10	330	4.3		10	330
n-Nitroso-di-n-Propylamine	621-64-7	10	330	1.8		10	330
Hexachloroethane	67-72-1	10	330	2.2		10	330
Nitrobenzene	98-95-1	10	330	0.9		10	330
Isophorone	78-59-1	10	330	1.1		10	330
2-Nitrophenol	88-75-5	10	330	1.6		10	330
2,4-Dimethylphenol	105-67-9	10	330	1.9		10	330
Benzoic Acid	65-85-0	NA	NA			50	1600
bis(2-Chloroethoxy) Methane	111-91-1	10	330	1.2		10	330
2,4-Dichlorophenol	120-83-2	10	330	1.7		10	330
1,2,4-Trichlorobenzene	120-82-1	10	330	2.3		10	330
Naphthalene	91-20-3	10	330	1.1		10	330
4-Chloroaniline	106-47-8	10	330	1.8		20	670
Hexachlorobutadiene	87-68-3	10	330	2.3		10	330
4-Chloro-3-Methylphenol	59-50-7	10	330	1.8		20	670
2-Methylnaphthalene	91-57-6	10	330	2.1		10	330
Hexachlorocyclopentadiene	77-47-4	10	330	1.9		10	330
2,4,6-Trichlorophenol	88-06-2	10	330	1.7		10	330
2,4,5-Trichlorophenol	95-95-4	25	800	1.3			

TABLE KN-5-5
KNOXVILLE LABORATORY
Semivolatile Organics
Contract Required Detection Limits (CRDL)[1], Method Detection Limits (MDL)[2], and Practical Quantitation Limits (PQL)[3]

Analyte	CAS Number	Water CRDL (ug/L)	Soil CRDL[4] (ug/Kg)	Water MDL (ug/L)	Soil MDL[4] (ug/Kg)	Water PQL (ug/L)	Soil PQL[4] (ug/kg)
2-Chloronaphthalene	91-58-7	10	330	1.1		10	330
2-Nitroaniline	88-74-4	25	800	1.4		50	1600
Dimethyl Phthalate	131-11-3	10	330	3.2		10	330
Acenaphthylene	208-96-8	10	330	1.4		10	330
2,6-Dinitrotoluene	606-20-2	10	330	1.2		10	330
3-Nitroaniline	99-09-2	25	800	1.6		50	1600
Acenaphthene	83-32-9	10	330	1.5		10	330
2,4-Dinitrophenol	51-28-5	25	800	2.7		50	1600
4-Nitrophenol	100-02-7	25	800	1.4		50	1600
Dibenzofuran	132-64-9	10	330	1.0		10	330
2,4-Dinitrotoluene	121-14-2	10	330	1.6		10	330
Diethylphthalate	84-66-2	10	330	4.4		10	330
4-Chlorophenyl-phenylether	7005-72-36	10	330	1.0		10	330
Fluorene	86-73-7	10	330	1.1		10	330
4-Nitroaniline	100-01-6	25	800	1.8		50	1600
4,6-Dinitro-2-Methylphenol	534-52-1	25	800	3.3		50	1600
n-Nitrosodiphenylamine	86-30-6	10	330	1.2		10	330
4-Bromophenyl-phenylether	101-55-3	10	330	1.2		10	330
Hexachlorobenzene	118-74-1	10	330	1.2		10	330
Pentachlorophenol	87-86-5	25	800	2.6		50	1600
Phenanthrene	85-01-8	10	330	1.1		10	330
Anthracene	120-12-7	10	330	1.1		10	330
Carbazole	86-74-8	10	330	NA		10	330
Di-n-Butylphthalate	84-74-2	10	330	3.4		10	330
Fluoranthene	206-44-0	10	330	1.1		10	330
Pyrene	129-00-0	10	330	1.4		10	330
Butylbenzylphthalate	85-68-7	10	330	3.0		10	330
3,3'-Dichlorobenzidine	91-94-1	10	330	1.6		20	670

TABLE KN-5-5
KNOXVILLE LABORATORY
Semivolatile Organics
Contract Required Detection Limits (CRDL)[1], Method Detection Limits (MDL)[2], and Practical Quantitation Limits (PQL)[3]

Analyte	CAS Number	Water CRDL (ug/L)	Soil CRDL[4] (ug/Kg)	Water MDL (ug/L)	Soil MDL[4] (ug/Kg)	Water PQL (ug/L)	Soil PQL[4] (ug/kg)
Benzo(a)anthracene	56-55-3	10	330	1.1		10	330
Chrysene	218-01-9	10	330	0.8		10	330
bis(2-Ethylhexyl)phthalate	117-81-7	10	330	1.1		10	330
Di-n-Octylphthalate	117-84-0	10	330	1.3		10	330
Benzo(b)fluoranthene	205-99-2	10	330	2.0		10	330
Benzo(k)fluoranthene	207-08-9	10	330	1.8		10	330
Benzo(a)pyrene	50-32-8	10	330	1.3		10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330	1.3		10	330
Dibenzo(a,h)anthracene	53-70-3	10	330	1.2		10	330
Benzo(g,h,i)perylene	191-24-2	10	330	1.2		10	330
1,2-Diphenylhydrazine	122-66-7			1.1			
Aniline	62-53-3			2.2			

[1] CRDLs apply to analyses performed in accordance with the USEPA CLP Statement of Work for Organics Analysis, Document Number OLM01.8.

[2] The Method Detection Limit for water was performed by SW-846 Method 8270 on 1-19-93.

[3] Practical Quantitation Limits are taken from SW-846 Method 8270; soil detection limits are based on wet weight, and are the same with and without GPC cleanup.

[4] Quantitation limits listed are for low soil procedure, and are based on wet weight. The quantitation limits based on dry weight as required, will be higher. The quantitation limits for medium level soil procedures will be higher by a factor of 30 than the low soil limits.

TABLE KN-5-6
KNOXVILLE LABORATORY
Pesticides and PCBs
Contract Required Detection Limits (CRDL)[1], Method Detection Limits (MDL)[2], and Practical Quantitation Limits (PQL)[3]

Analyte	CAS Number	Water CRDL (ug/L)	Soil CRDL[4] (ug/Kg)	Water MDL (ug/L)	Soil MDL[4] (ug/Kg)	Water PQL (ug/L)	Soil PQL[4] (ug/Kg)
α-BHC	319-84-6	0.05	1.7	0.0005		0.03	2
β-BHC	319-85-7	0.05	1.7	0.0025		0.06	4
δ-BHC	319-86-8	0.05	1.7	0.0054		0.09	6
γ-BHC (Lindane)	58-89-9	0.05	1.7	0.0007		0.04	3
Heptachlor	76-44-8	0.05	1.7	0.0017		0.03	2
Aldrin	309-00-2	0.05	1.7	0.0018		0.04	2
Heptachlor Epoxide	1024-57-3	0.05	1.7	0.0023		0.8	60
Endosulfan I	959-98-8	0.05	1.7	0.0041		0.1	9
Dieldrin	60-57-1	0.10	3.3	0.0016		0.02	2
4,4'-DDE	72-55-9	0.10	3.3	0.0013		0.04	2
Endrin	72-20-8	0.10	3.3	0.0038		0.06	4
Endosulfan II	33213-65-9	0.10	3.3	0.0035		0.04	2
4,4'-DDD	72-54-8	0.10	3.3	0.0020		0.1	8
Endosulfan sulfate	1031-07-8	0.10	3.3	0.0060		0.7	40
4,4'-DDT	50-29-3	0.10	3.3	0.0018		0.1	8
Methoxychlor	72-43-5	0.50	17.0	0.0091		2	120
Endrin Aldehyde	7421-93-4	0.10	3.3			0.2	16
Endrin Ketone	53494-70-5	0.10	3.3			NA	NA
α-Chlordane	5103-71-9	0.05	1.7			NA	NA
γ-Chlordane	5103-74-2	0.05	1.7			NA	NA
Chlordane (Tech)	57-74-9			0.013		0.1	9
Toxaphene	8001-35-2	5.0	170.0	0.43		2	160
Aroclor 1016	12674-11-2	1.0	33.0			0.6	40
Aroclor 1221	11104-28-2	2.0	67.0			0.6	40
Aroclor 1232	11141-16-5	1.0	33.0			0.6	40
Aroclor 1242	53469-21-9	1.0	33.0	0.041		0.6	40
Aroclor 1248	12672-29-6	0.50	33.0			0.6	40

TABLE KN-5-6
KNOXVILLE LABORATORY
Pesticides and PCBs
Contract Required Detection Limits (CRDL)[1], Method Detection Limits (MDL)[2], and Practical Quantitation Limits (PQL)[3]

Analyte	CAS Number	Water CRDL (ug/L)	Soil CRDL[4] (ug/Kg)	Water MDL (ug/L)	Soil MDL[4] (ug/Kg)	Water PQL (ug/L)	Soil PQL[4] (ug/Kg)
Aroclor 1260	11096-82-5	1.0	33.0			1	90

1 CRDLs apply to analyses performed in accordance with the USEPA Statement of Work for Organics Analysis, Document Number OLM01.8.

2 The Method Detection Limit studies for pesticides/PCBs were performed on 7/29/93 using SW-846 method 8080.

3 Practical Quantitation Limits are based on SW-846 method 8080.

4 Quantitation limits listed for soil are based on wet weight. The quantitation limits based on dry weight as required, will be higher.

TABLE KN-5-7
KNOXVILLE LABORATORY
Explosives
Method Detection Limits (MDL)[1] and Practical Quantitation Limits (PQL)[2]

Analyte	CAS Number	Water MDL (ug/L)	Soil MDL (mg/kg)	Water PQL (ug/L)	Soil PQL (mg/kg)
HMX	2691-41-0	4.8		25	2.2
RDX	121-82-4	5.4		24	1.0
1,3,5-Trinitrobenzene	99-35-4	3.3		24	0.25
1,3-Dinitrobenzene	99-65-0	2.4		24	0.25
Tetryl	479-48-8	5.9		26	0.65
Nitrobenzene	98-95-3	1.0		28	0.26
2,4,6-Trinitrotoluene	118-96-7	0.0		24	0.26
2,4 and 2,6-Dinitrotoluene	121-14-2 and 606-20-2	9.4		25	0.25
2-Nitrotoluene	88-72-2	5.1		25	0.25
4-Nitrotoluene	99-99-0	15		24	0.25
3-Nitrotoluene	99-08-1	3.6		28	0.25

[1] The Method Detection Limit study was performed on 11/20/93 using SW-846 method 8330.

[2] Practical Quantitation Limits are based on SW-846 method 8330.

TABLE KN-5-8
KNOXVILLE LABORATORY
Aromatic Volatiles/Low Boiling Petroleum Hydrocarbons by GC
Method Detection Limits (MDL)[1], and
Practical Quantitation Limits (PQL)

Analyte	CAS Number	Water MDL (ug/L)	Soil MDL (mg/Kg)	Water PQL (ug/L)	Soil PQL (mg/Kg)
Benzene	71-43-2	0.12	0.01	1	0.1
Chlorobenzene	108-90-7	0.04	0.01	1	0.1
1,4-Dichlorobenzene	106-46-7	0.05	0.01	1	0.1
1,3-Dichlorobenzene	541-73-1	0.05	0.01	1	0.1
1,2-Dichlorobenzene	95-50-1	0.05	0.01	1	0.1
Ethylbenzene	100-41-4	0.04	0.01	1	0.1
Toluene	108-88-3	0.10	0.02	1	0.1
m,p-Xylene		0.11	0.05	1	0.1
o-Xylene	95-47-6	0.06	0.02	1	0.1
Methyl-*tert*-butyl ether		0.12	0.02	1	0.1
Low Boiling Petroleum Hydrocarbons		7.6	1.7	50	5.0

[1] MDLs were determined on 1/6/93 for water and on 1/7/93 for soil samples using SW-846 methods 8020/CA MOD. 8015 in series.

TABLE KN-5-9
KNOXVILLE LABORATORY
Polynuclear Aromatic Hydrocarbons by HPLC
Method Detection Limits (MDL)[1], and
Practical Quantitation Limits (PQL)[2]

Analyte	CAS Number	Water MDL (µg/L)	Soil MDL (µg/kg)	Water PQL (µg/L)	Soil PQL (µg/kg)
Naphthalene	91-20-3	0.016		0.16	16
Acenaphthylene	208-96-8	0.015		0.16	16
Acenaphthene	83-32-9	0.015		0.16	16
Fluorene	86-73-7	0.015		0.080	8.0
Phenanthrene	85-01-8	0.014		0.080	8.0
Anthracene	120-12-7	0.018		0.080	8.0
Fluoranthene	206-44-0	0.031		0.080	8.0
Pyrene	129-00-0	0.022		0.080	8.0
Benzo(a)anthracene	56-55-3	0.013		0.080	8.0
Chrysene	218-01-9	0.018		0.080	8.0
Benzo(b)fluoranthene	205-99-2	0.012		0.080	8.0
Benzo(k)fluoranthene	207-08-9	0.015		0.080	8.0
Benzo(a)pyrene	50-32-8	0.012		0.080	8.0
Dibenzo(ah)anthracene	53-70-3	0.023		0.080	8.0
Benzo(ghi)perylene	191-24-2	0.033		0.080	8.0
Indeno(123-cd)pyrene	193-39-5	0.037		0.080	8.0

[1] MDLs were determined on 9/16/93 for waters using SW-846 method 8310.

[2] PQLs are based on SW-846 method 8310.

TABLE KN-5-10
KNOXVILLE LABORATORY
Total High Boiling Petroleum Hydrocarbons
Method Detection Limits (MDL)[1]
and Practical Quantitation Limits (PQL)

Analyte	CAS Number	Water MDL (ug/L)	Soil MDL (mg/kg)	Water PQL (ug/L)	Soil PQL (mg/kg)
Total high boiling petroleum hydrocarbons, as compared to diesel fuel		19		50	5

[1] The Method Detection Limit study was performed on 1/9/93 using CA MOD. method 8015.

TABLE KN-5-11
KNOXVILLE LABORATORY
Halogenated and Aromatic Volatiles by GC
Method Detection Limits $(MDL)_1$
and Practical Quantitation Limits $(PQL)_2$

Analyte	CAS Number	Water MDL (ug/L)	Soil MDL (µg/kg)	Water PQL (ug/L)	Soil PQL (µg/kg)
Dichlorodifluoromethane	75-71-8	0.50		1.8	1.8
Chloromethane	74-87-3	0.26		0.8	0.8
Vinyl chloride	75-01-4	0.37		1.8	1.8
Bromomethane	74-83-9	0.31		1.2	1.2
Chloroethane	75-00-3	0.34		5.2	5.2
Trichlorofluoromethane	75-69-4	0.47		2.0	2.0
1,1-Dichloroethene	75-35-4	0.20		1.3	1.3
Methylene Chloride	75-09-2	1.3		15	15
trans-1,2-Dichloroethene	156-60-5	0.09		1.0	1.0
1,1-Dichloroethene	75-34-3	0.09		0.7	0.7
cis-1,2-Dichloroethene	156-59-2	0.10		1.0	1.0
Trichloromethane	67-66-3	0.06		0.5	0.5
1,1,1-Trichloroethane	71-55-6	0.14		0.3	0.3
Carbon Tetrachloride	56-23-5	0.14		1.2	1.2
Benzene	71-43-2	0.10		2.0	2.0
1,2-Dichloroethane	107-06-2	0.09		0.3	0.3
Trichloroethene	79-01-6	0.14		1.2	1.2
1,2-Dichloropropane	78-87-5	0.06		0.4	0.4
Bromodichloromethane	75-27-4	0.05		1.0	1.0
Dibromomethane	74-95-3			2.0	2.0
2-Chloroethylvinyl Ether	110-75-8	0.21		1.3	1.3
cis-1,3-Dichloropropene	10061-01-5	0.04		3.4	3.4
Toluene	108-88-3	0.08		2.0	2.0
trans-1,3-Dichloropropene	10061-01-5	0.05		3.4	3.4
1,1,2-Trichloroethane	79-00-5	0.09		0.2	0.2
Tetrachloroethene	127-18-4	0.16		0.3	0.3
Dibromochloromethane	124-48-1	0.12		0.9	0.9

TABLE KN-5-11
KNOXVILLE LABORATORY
Halogenated and Aromatic Volatiles by GC
Method Detection Limits (MDL)[1] and
Practical Quantitation Limits (PQL)[2]
(continued)

Analyte	CAS Number	Water MDL (ug/L)	Soil MDL (µg/kg)	Water PQL (ug/L)	Soil PQL (µg/kg)
Dichlorodifluoromethane	75-71-8	0.50		1.8	1.8
n-Hexyl Chloride	544-10-8			1.0	1.0
Chlorobenzene	108-90-7	0.07		2.0	2.0
Ethyl Benzene	100-41-4	0.11		2.0	2.0
1,1,1,2-Tetrachloroethane	630-20-6			0.3	0.3
Xylenes (total)	1330-20-7	0.10		1.0	1.0
Bromoform	75-25-2	0.11		2.0	2.0
1,1,2,2-Tetrachloroethane	79-34-5	0.16		0.3	0.3
1,2,3-Trichloropropane	96-18-4	0.16		1.0	1.0
Phenyl Bromide (Bromobenzene)	108-86-1			2.0	2.0
2-Chlorotoluene	108-41-8			1.0	1.0
1,3-Dichlorobenzene	541-73-1	0.06		3.2	3.2
1,4-Dichlorobenzene	106-46-7	0.09		2.4	2.4
1,2-Dichlorobenzene	95-50-1	0.07		1.5	1.5
bis(2-Chloroisopropyl) ether	108-60-1			20	20

[1] The Method Detection Limit Study was performed on 12/11/92 using SW-846 methods 8010 and 8020 in series.

[2] Practical quantitation limits are based on SW-846 methods 8010 and 8020.

APPENDIX

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PERFORMANCE EVALUATION SAMPLE STUDIES

TABLE 6-1
KNOXVILLE LABORATORY
Performance Evaluation Sample Studies

PE Sample Program Description	Analysis Performed	Frequency of Participation
US EPA Water Supply Study	Res. Cl, SO_4, Turbidity, Anions, PAH by 8310, VOC, THM, Pesticides, Herbicides, Corrosivity, Sodium, Trace Metals, Cyanide (CN).	Semiannual
US EPA Water Pollution Study	Trace Metals, Minerals, Nutrient, Demand, PCBs, Chlorinated Hydrocarbon, Pesticides, Volatile Halocarbons, Volatile Aromatics, Total CN, TSS, Oil and Grease, Phenols, Res. Cl.	Semiannual
US EPA Contract Laboratory Program - Organics	CLP Organics	Quarterly
US Army Corps of Engineers	Explosives, Volatile Organics, Halogenated VOAs, Aromatic VOAs, Semivolatile Organics, Pesticides, PCBs, PP and RCRA Metals, TOC, CN, Anions, Herbicides.	Biennial
New York State Potable and Non Potable Water Studies	Refer to EPA WS and WP analyses.	Semiannual
State of North Carolina Department of Human Resources	MBAS, Turbidity, COD, Semivolatile Organics.	Annual

APPENDIX

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PREVENTIVE MAINTENANCE SCHEDULES

TABLE 7-1
KNOXVILLE LABORATORY
Maintenance Schedule

Graphite Furnace Atomic Absorption[1]			
Daily	**Monthly**	**Semi-annually**	**Annually**
Clean Optical Windows	Check coolant level in cooling unit. Add coolant if error message appears	Replace contact cylinders	Notify repairman to perform cleaning of optics
Clean contact cylinders			
Check tubes and platform, replace if corroded, flaking, or if low absorbance results			

[1] Refer to manufacturer's instructions for each instrument to perform maintenance operations

TABLE 7-2
KNOXVILLE LABORATORY
Maintenance Schedule

ICP[1]		
Daily	**Monthly**	**Semi-annually**
Check the pump winding, replace if needed.	Clean intelligence controller filters	Change vacuum pump oil
	Check and clean nebulizer, mixing chamber and torch.	
	Check the pump capillary tubing, clean or replace if needed.	
	Clean optical path windows	
	Clean RF generator filters	

[1] Refer to manufacturer's instructions for each instrument to perform maintenance operations.

TABLE 7-3
KNOXVILLE LABORATORY
Maintenance Schedule

High Pressure Liquid Chromatography[1]
As Needed
Column change: Columns are replaced when peak shape and resolution indicate that the chromatographic performance of the column is below method requirements.
Filters are cleaned every time a column change takes place.
The slides on the auto sampler are oiled when the sample does not advance as required.
[1] refer to the manufacturer's instructions for each instrument to perform maintenance operations.

TABLE 7-4
KNOXVILLE LABORATORY
Maintenance Schedule

Ion Chromatography[1]				
As Needed	**Daily**	**Weekly**	**Monthly**	**Semi-annually**
Clean micromembrane suppressor when decreases in sensitivity are observed	Check plumbing	Check pump heads for leaks	Clean conductivity cell	Replace gradient pump piston seal
Change fuses when power problems occur	Check UV/VIS detector for leaks	Check filter (inlet)	Check conductivity cell for calibration	Clean high pressure valve
Change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated.			Check all air and liquid lines for discoloration and crimping, if indicated.	
Degas pump head when flow is erratic				

[1] Refer to manufacturer's instructions for each instrument to perform maintenance operations.

TABLE 7-5
KNOXVILLE LABORATORY
Maintenance Schedule

Total Organic Halide Analyzer[1]		
Daily	**Weekly**	**Monthly**
Check exit tube and quartz wool.	Change quartz wool in inlet liner and exit tube.	Examine and clean pyrolysis tube.
Check for gas bubbles in titration sidearm.	Measure gas flow.	Clean titration cell.
Check inlet area for GAC spills	Perform cell performance check.	Perform electronic test.
Check electrolyte level, add if needed		Check o-rings, replace if worn.
Clean quartz boat.		

[1] Refer to manufacturer's instructions for each instrument to perform maintenance operations.

TABLE 7-6
KNOXVILLE LABORATORY
Maintenance Schedule

Total Organic Carbon Analyzer[1]			
Daily	**Weekly**	**Bi-monthly**	**Monthly**
Check oxygen supply	Check liquid-flow-rate-pump-tubing conditions	Change pump tubing	Change Sn and Cu.
Check persulfate supply	Check for moisture in LiOH tube.		
Check acid supply	Check injection port septum.		
Check printer paper.			
Check Sn and Cu scrubber			
Add a few drops of H_3PO_4 to GLS.			
Check carrier gas flow rate (≈ 200 cc/min).			

(1) Refer to manufacturer's instructions for each instrument to perform maintenance operations.

TABLE 7-7
KNOXVILLE LABORATORY
Maintenance Schedule

Gas Chromatography[1]	
As Needed	**Semi-annually**
Replace front portion of column packing or break off front portion of capillary columns. Replace column if this action fails to restore performance of the column or when column performance (*e.g.* peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.	Perform ECD wipe test.
Change glass wool plug in injection port and/or replace injection port liner with cleaned liner when front portion of column packing is changed or front portion of capillary column is removed.	Replace carbon filters at instrument effluent sites.
Replace septum (usually approximately every 100 injections).	
Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).	
replace or repair flow controller if constant gas flow cannot be maintained.	
Change fuse when performance indicates fuse has blown.	
Reactivate external carrier gas dryers when blue indicator changes to pink.	
Clean detectors when baseline indicates contamination or when response is low.	
Reactivate flow controller filter dryers when presence of moisture is suspected.	
Tekmar purge and trap devices: replace traps and columns when poor response or disappearance of reactive or poorly trapped compounds.	

[1] Refer to manufacturer's instructions for each instrument to perform maintenance operations.

TABLE 7-8
KNOXVILLE LABORATORY
Maintenance Schedule

Mass Spectrometers[1]			
Instrument	As Needed	Monthly	Annually
Mass Spectrometer	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between service contract maintenance.	Not Applicable	Not Applicable
	Electron multiplier: replace when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.		
	Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.		
Neslab Chiller	Check coolant level. Add coolant as required to maintain cooling.	Vacuum outside chiller to prevent dust from clogging filters	Clean on-line filter in Neslab

[1] Refer to manufacturer's instructions for each instrument to perform maintenance operations

APPENDIX

KNOXVILLE - 8

ADDITIONAL OPERATION-SPECIFIC INFORMATION

UNCONTROLLED COPY

QUALITY ASSURANCE MANAGEMENT PLAN

Revision 2
June 1994

Quality Assurance Manager — Date: 06-07-94

Laboratory Director — Date: 6-7-94

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ACRONYMS AND ABBREVIATIONS

AAS	atomic absorption spectroscopy
ACOE	Army Corps of Engineers
APHA	American Public Health Association
ANSI	American National Standards Institute
ASME	Americas Society of Mechanical Engineers
ASQC	American Society for Quality Control
ASTM	American Society for Testing and Materials
BFB	p-bromofluorobenzene
CCV	continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLP	Contract Laboratory Program
CPM	counts per minute
CWA	Clean Water Act
DER	duplicate error ratio
DFTPP	decafluorotriphenylphosphine
DOE	Department of Energy
DQO	data quality objective
EICP	extracted ion current profile
EML	Environmental Measurement Laboratory
EPA	U.S. Environmental Protection Agency
FIFRA	Federal Insecticide, Fungicide, and Rodenticide Act
FOM	figure of merit
FS	feasibility study
FT-IR	Fourier transform infrared spectrophotometry
FWHM	full width half maximum
GC	gas chromatograph
GC-MS	gas chromatograph-mass spectroscopy
GFAA	graphite furnace atomic absorption
GLP	good laboratory practice
GMP	good measurement practice
GPC	gas proportional counter
HEPA	high efficiency particular air
HPLC	high performance liquid chromatography
IC	ion chromatograph
ICP-AES	inductively coupled plasma/atomic emission spectroscopy
ICV	initial calibration verification
IDL	instrument detection limit
LAS	Lockheed Analytical Services
LCS	laboratory control sample
LDMS	laboratory data management system
LDR	linear dynamic range
LESAT	Lockheed Environmental Systems & Technologies Company
LLD	lower limit of detection
MDA	minimum detectable activity

(Continued...)

ACRONYMS AND ABBREVIATIONS *(Continued)*

MDL	method detection limit
MS	matrix spike
MSA	method of standard additions
MSD	matrix spike duplicate
NCAR	Nonconformance & Corrective Action Record
NEIC	National Enforcement Investigations Center
NIST	National Institute for Standards and Technology
NRC	Nuclear Regulatory Commission
OSHA	Occupational Safety and Health Administration
PE	performance evaluation
QA	quality assessment
QAMP	Quality Assurance Management Plan
QAMS	Quality Assurance Management Staff
QC	quality control
QCCS	quality control check standard
RCRA	Resource Conservation and Recovery Act
RDL	reporting detection limit
RER	replicate error ratio
RI	remedial investigation
RPD	relative percent difference
RRF	relative response factor
RRT	relative retention time
RSD	relative standard deviation
SARA	Superfund Amendments and Reauthorization Act
SDG	sample delivery group
SDWA	Safe Drinking Water Act
SOP	standard operating procedure
SOW	statement of work
SRM	standard reference material
TQM	Total Quality Management
TSCA	Toxic Substances Control Act
UPS	uninterruptable power supply
VOA	volatile organic analysis
VOC	volatile organic constituent
VTSR	validated time of sample receipt
WP	water pollution
WS	water supply

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CHAPTER 1
INTRODUCTION

This manual is an overview of the Quality Assurance Program that governs all Lockheed Analytical Services (LAS) operations. It outlines the purpose, policies, organization, responsibilities, and operations related to ensuring high-quality performance in all LAS activities. The manual is intended to provide guidance to management, project leads, laboratory analysts, and support personnel in the uniform implementation of general quality assurance requirements specific to LAS by providing a minimum set of quality management elements required to provide analytical products and services at LAS. The project-specific requirements delineated in project plans may supersede the general quality requirements described in this manual. It is essential that all LAS personnel be familiar with the policies, objectives, and procedures outlined in this management plan so that they fully understand their roles and responsibilities in the overall LAS Quality Assurance Program. Furthermore, all subcontractors employed by LAS must adhere to the set of QA requirements delineated in this manual.

1.1 POLICY

The preparation of this management plan and implementation of the quality assurance philosophy and procedures specified herein are in accordance with Lockheed Corporate quality assurance policy. Corporate regulations and guidelines require the implementation of quality assurance activities and the maintenance of sufficient documentation to demonstrate the generation of legally defensible environmental data. Lockheed corporate policies on quality are based on the following concepts:

- To achieve the mission, goals, and long-term objectives of the Corporation, we must provide our clients with products and services that satisfy their definitions and expectations of quality.

- To achieve the required quality in these products and services at a competitive price, a strategy to obtain that level of quality is necessary.

- The pursuit of quality, and its improvement, is a continuous process with measurable objectives.

- Each employee is a customer and a supplier; each is personally responsible and accountable for the quality of his or her own work.

It is the policy of Lockheed Environmental Systems & Technologies Company (LESAT) to provide products and services to its internal and external customers that satisfy or exceed their quality requirements and expectations. LAS quality policy is to produce analytical data reports that meet client and regulatory requirements, are useable for their intended purpose, are technically correct, and are produced within contract specifications. In addition, it is our corporate, company, and division policy to continually improve our procedures, products, and services. Lockheed management fully supports continuous quality improvement efforts to reduce cost without compromising quality.

Lockheed stresses the importance of quality at every level in the Corporation from the Chief

Executive Officer to the individual employee. LAS management has made a sustained commitment of personnel and resources to develop, implement, assess, and continually improve our technical and management operations. Only with management's full participation is it possible to instill this commitment to excellence in all LAS employees.

1.2 GOALS

The primary goal of the LAS Quality Assurance Program is to ensure that all measurement data generated are scientifically and legally defensible, of known and appropriate quality, and thoroughly documented so that they provide sound support for environmental decisions. A supporting goal is to comply with all environmental regulations established by local, state, and federal regulatory authorities.

The specific goals are:

- To provide a uniform framework for generating physical and chemical data.
- To operate under a comprehensive, effective, ongoing quality assurance program that focuses on preventive maintenance, which will help ensure the timely and effective completion of each measurement effort.
- To instill a commitment to quality assurance and individual excellence at all levels of the organization.
- To assist in the early detection of anomalies and nonconformances that might adversely affect data quality.
- To establish the quality assurance objectives for the measurement systems and to assess and monitor analytical data quality in terms of precision, accuracy, representativeness, comparability, completeness, and detectability through the use of proven methods.
- To establish procedures to demonstrate, through the use of control charts and other means, that analytical systems are in a state of statistical control.
- To enable personnel responsible for the production of the data to identify and implement corrective actions necessary to ensure data integrity.
- To ensure that the appropriate type and degree of quality control are applied during an analytical run.
- To ensure adequate document control.
- To eliminate data anomalies through the implementation of an automated, efficient data-handling and data-validation system.
- To develop and follow good laboratory practices (GLPs), good measurement practices (GMPs), and standard operating procedures (SOPs).
- To provide sufficient flexibility for implementing customized quality assurance procedures to meet customers' specific requirements for data quality.
- To establish guidelines for adequate control of procurement of instruments, chemicals, and services.
- To ensure proper tracking of samples and analytical data by implementing an automated laboratory data management system (LDMS).

- To ensure that computer hardware and software used in producing analytical data are independently validated and documented according to the intended use of the software.

1.3 KEY PROGRAM ELEMENTS

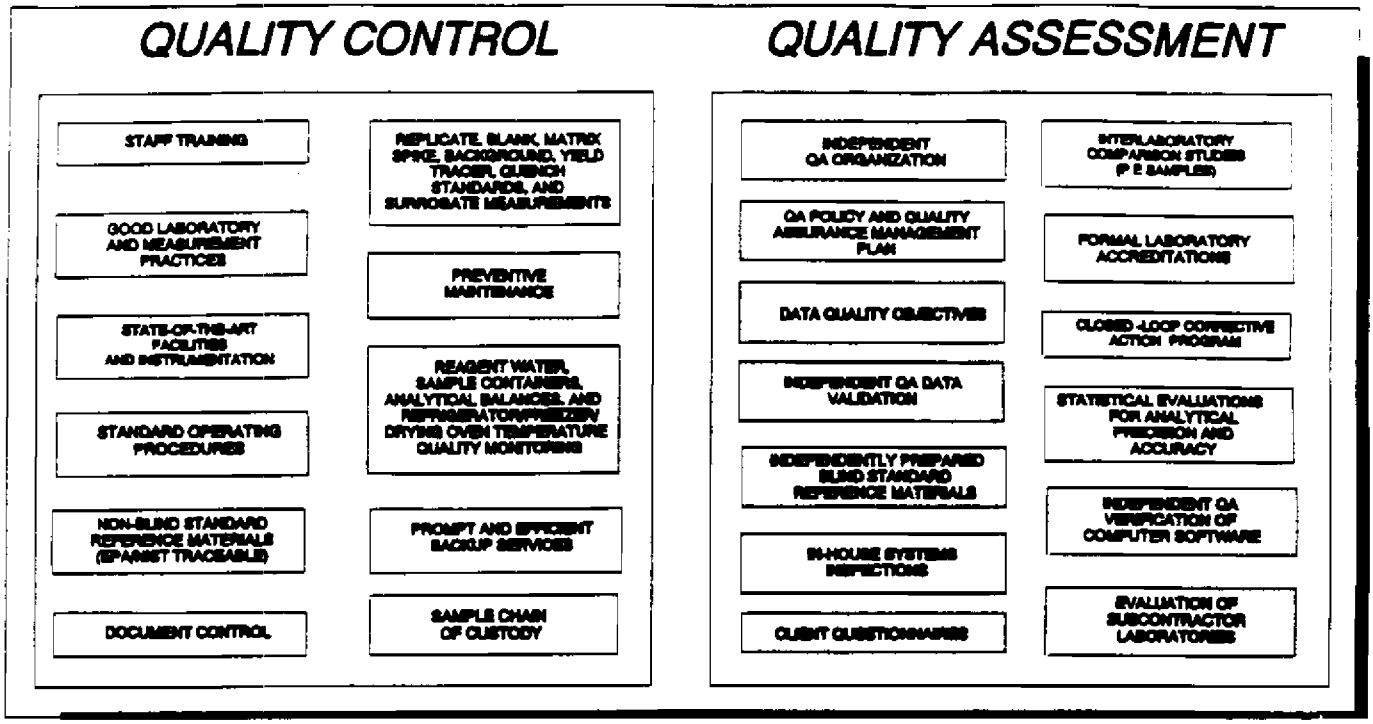
Formalizing and implementing sound quality assurance procedures are the initial steps in ensuring that data quality is within specified control limits and is well documented. The LAS Quality Assurance Program provides a quantitative approach to ensuring the integrity of all data generated by the laboratory staff. Key elements of the program, shown in Figure 1, help ensure that all analytical data meet customer, LAS, and regulatory requirements. Through application of these elements, the program monitors LAS performance in relation to quality assurance objectives and quality control requirements identified for each analytical method. This approach incorporates the proper control, assessment, and documentation of analytical data quality.

1.4 SUPPORTING DOCUMENTS

The LAS Quality Assurance Program is responsive to, and in compliance with, the guidelines and specifications described in the following documents:

- U.S. Environmental Protection Agency (EPA) Quality Assurance Management Staff (EPA QAMS-005/80).
- American National Standard Institute/American Society for Quality Control (ANSI/ASQC-E4-19xx).
- American National Standard Institute/American Society for Quality Control (ANSI/ASQC Q94-1987; ISO 9004).
- International Standard ISO/IEC Guide 25.
- U.S. Department of Energy (DOE) Order 5700.6C "Quality Assurance."
- American Society of Mechanical Engineers (ASME) NQA-1 (1989 Edition).
- Test Methods for Evaluating Solid Waste (EPA SW-846 Third Edition, 1986 and current updates).
- Methods for Chemical Analysis of Water and Wastes (EPA-600/4-79-020).
- Current revisions of the EPA Contract Laboratory Program (CLP) Statements of Work (SOW) for inorganic and organic analyses.
- Handbook for Analytical Quality Control in Water and Wastewater Laboratories (EPA-600/ 4-79-019).
- Good Laboratory Practice Standards (40 CFR Part 792).
- Standard Methods for the Examination of Water and Wastewater (18th Edition, 1992).
- Annual Standards Books of the American Society for Testing and Materials (ASTM).
- EPA Prescribed Procedures for Measurement of Radioactivity in Drinking Water (EPA-600/ 4-80/032).
- EPA Radiochemical Analytical Procedures for Analysis of Environmental Samples (EPA-LV-0539-17).

Figure 1. Key Elements of the LAS Quality Assurance Program



- EPA Eastern Environmental Radiation Facility Radiochemistry Procedures Manual (EPA/520/5-84/006).

- EPA Procedures for Radiochemical Analysis of Nuclear Reactor Aqueous Solutions (R4-73-0144).

- HASL Environmental Measurements Laboratory Procedures Manual (HASL-300).

Furthermore, the LAS Quality Assurance Program follows standards and requirements mandated by the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA); the Resource Conservation and Recovery Act (RCRA); the Superfund Amendments and Reauthorization Act (SARA); the Toxic Substances Control Act (TSCA); the Nuclear Regulatory Commission (NRC); and the Occupational Safety and Health Administration (OSHA).

1.5 DEFINITIONS

The terms quality assurance, quality assessment, and quality control appear frequently in this plan and are central to the themes presented. These terms are defined as follows:

Quality assurance is the total, integrated program for ensuring the integrity of measurement data to support environmental decisions and potential litigation challenges. It consists of two separate but related activities: quality assessment and quality control.

Quality assessment (QA) is the overall system of activities enacted to ensure that the quality control activities are effective. It involves continuously evaluating the performance of the data, the production system, and the quality of the analytical data generated.

Quality control (QC) is the routine application of procedures to control the quality of

measurement data and to ensure that data meet the needs of the customer. QC procedures are used to maintain a measurement process in a state of statistical control; that is, a state in which the analytical system is stable and data are reproducible within defined limits. The aim of QC is to provide data of a quality that is satisfactory, adequate, dependable, and economical.

1.6 CONFORMANCE TO NATIONALLY RECOGNIZED STANDARDS

This plan addresses the quality assurance requirements outlined in U.S. EPA QAMS-005/80, ANSI/ASQC-E4-19xx, DOE Order 5700.6C (1991 edition), ANSI/ASQC Q94 (ISO 9004), and NQA-1. Table 1 identifies the sections of this plan and the SOPs in which requirements are addressed. The Quality Assurance Management Plan (QAMP) is supported by SOPs, which are revised routinely to reflect LAS current operations. Procedures described in the SOPs that are revised following this revision of the QAMP supersede the requirements specified in this document. References to SOPs that detail the specific elements of the LAS Quality Assurance Program are also provided in each section of the manual. A complete index of the SOPs is presented in Appendix C and is current as of the publication date of this manual.

Table 1. Sections in the LAS Quality Assurance Management Plan and Standard Operating Procedures that address EPA, NQA-1, ANSI/ASQC, DOE, and ISO Requirements (Page 1 of 3)

ASME NQA-1 Requirements	QAMP Section (SOP#)	EPA QAMS-005 Requirements	QAMP Section (SOP#)
Organization	2	Title Page	Title Page
QA Program	1	Table of Contents	Table of Contents
Design Control	3	Project Description	1
Procurement Document Control	16 (232,284)	Project Organization and Responsibility	2
Instructions, Procedures, and Drawings	5 (method SOPs)	QA Objectives	1,3 (method SOPs)
Document Control	10 (1,127)	Sampling Procedures	4(2,9,85,105,173)
Control of Purchased Items and Services	16 (232,284)	Sample Custody	4,13 (2,9)
Identification and Control of Items	15 (227,283)	Calibration Procedures and Frequency	5,6,7,8 (method SOPs)
Control of Processes	4,6,7,8,11,12 (method SOPs)	Analytical Procedures	5,6,7,8 (method SOPs)
Inspection	9 (10)	Data Analysis, Reduction, Validation	10,11,12
		and Reporting	(8,12,13,88,278)
Test Control	6,7,8 (method SOPs)	Internal QC Checks and Frequency	6,7,8 (method SOPs)
Control of Measuring and Test Equipment	14 (15,188)	Performance and System Audits	9 (10)
Handling, Storage, and Shipping	4,10,13 (2,9,85,105,173)	Preventative Maintenance	14 (15,188)
Inspection, Test, and Operating Status	9,11 (8,10)	Assessment of Precision, Accuracy and Completeness	12 (8,12,13,88)
Control of Nonconforming Items	15,16 (227,232,283,284)	Corrective Actions	15 (190)
Corrective Action	15 (190)	QA Reports to Management	17
Quality Assurance Records	10,11,13,17 (1,6,127)		
Audits	9 (10)		

Table 1. Sections in the LAS Quality Assurance Management Plan and Standard Operating Procedures that address EPA, NQA-1, ANSI/ASQC, DOE, and ISO Requirements (Page 2 of 3)

ANSI/ASQC E4-19xx Requirements	QAMP Section (SOP#)	DOE ORDER 5700.6C	QAMP Section (SOP#)
PART A: MANAGEMENT SYSTEMS		PART 1: MANAGEMENT	
Management and Organization	1,2	Program	1
Quality System and Description	1,2,3	Personnel Qualification and Training	2 (110)
Personnel Qualification and Training	2 (110)	Quality Improvement	1,3 (8,10,12,13,88,188,190)
Procurement of Items and Services	16 (232,284)	Documents and Records	10,17 (1,6,127)
Documents and Records	10,17 (1,6,127)	PART 2: PERFORMANCE	
Computer Hardware & Software	10 (123,124)	Work Processes	4,5,6,7,8,10, 11,12,13,14,15,16 (method SOPs & 2,9,227,283)
Planning	3,14,15	Design	N/A
Implementation of Work Processes	4,5,6,7,8 (method SOPs)	Procurement	16 (232,284)
Assessment and Response	9,11,12,17 (8,10,12,13,88)	Inspection and Acceptance Testing	4,6,7,8,9,11,14,17 (10,284)
Quality Improvement	1,3,6,7,8 (8,10,12,13,88,188,190)	PART 3: ASSESSMENT	
PART B: COLLECTION AND EVALUATION OF ENVIRONMENTAL DATA		Self/Management Assessment	9,11,12,15,17 (8,10,12,13,88)
Planning and Scoping	1,3,15	Independent Assessment	9,11,12 (8,10,12,13,88)
Design of Data Collection Operation	3		
Implementation of Planned Operations	4,5,6,7,8 (method SOP)		
Assessment and Response	9,11,12,17 (8,10,12,13,88)		
Assessment and Verification of Data Useability	N/A		
PART C: DESIGN, CONSTRUCTION, AND OPERATION OF ENGINEERED ENVIRONMENTAL SYSTEMS	N/A		

Table 1. Sections in the LAS Quality Assurance Management Plan and Standard Operating Procedures that address EPA, NQA-1, ANSI/ASQC, DOE, and ISO Requirements (Page 3 of 3)

ANSI/ASQC Q94 (ISO-9004) Requirements	QAMP Section (SOP#)
4. Management Responsibility	1
5. Quality System Principles	1,3
5.2 Structure of the Quality System	2
5.3 Documentation of the Quality System	1,2,3
5.4 Auditing the Quality System	9 (10)
6. Economics-Quality Related Cost Considerations	N/A
7. Quality In Marketing	N/A
8. Quality in Specification & Design	N/A
9. Quality in Procurement	16 (227,232,284)
10. Quality in Production	4,5,6,7,8,10 (method SOPs)
11. Control of Production	4,6,7,8,16 (method SOPs & 5.284)
11.2 Material Control & Traceability	4,16 (5,284)
12. Product Verification	6,7,8,9,11,12 (method SOPs)
13. Control of Measuring & Test Equipment	14 (15,188)
14. Non-Conformity	15 (190)
15. Corrective Action	15 (190)
16. Handling & Post Production Functions	4,10,13
17. Quality Documentation & Records	10 (1,6,127)
18. Personnel (Training)	2 (110)
19. Product Safety and Liability	1 (Safety Manual)
20. Use of Statistical Methods	6,7,8,12

CHAPTER 2
LABORATORY ORGANIZATION AND RESPONSIBILITIES

2.1 ORGANIZATIONAL STRUCTURE

The LAS organizational structure, shown in Figure 2, is designed to ensure that analytical operations are effective and cost-efficient. All levels of the laboratory staff are involved in implementing the formal LAS Quality Assurance Program; responsibilities of each staff level are described below.

It is Lockheed's policy to staff technical and quality assurance positions with personnel who have the education, training, and experience sufficient to competently accomplish their assigned duties. The LAS Director is responsible for ensuring that all personnel receive auxiliary training, as needed, to increase the understanding and skills that they apply to their positions.

2.2 AUTHORITY AND RESPONSIBILITY

The LAS management recognizes that the responsibility for a high-quality product starts with each employee; however, the ultimate responsibility for data and service quality and reliability resides with the Director. The LAS Director has appointed a Quality Assurance Manager to implement the LAS Quality Assurance Program and to provide continuous, independent oversight of laboratory operations. The Quality Assurance Manager is independent of daily laboratory activities and reports directly to the LAS Director to ensure unbiased evaluation of operations. The Quality Assurance Manager is the focal point for information on the LAS Quality Assurance Program and is responsible for providing advice and assistance to laboratory staff and management. The Quality Assurance Manager also has the authority to cease analytical activities that are out of control.

2.2.1 LAS Director

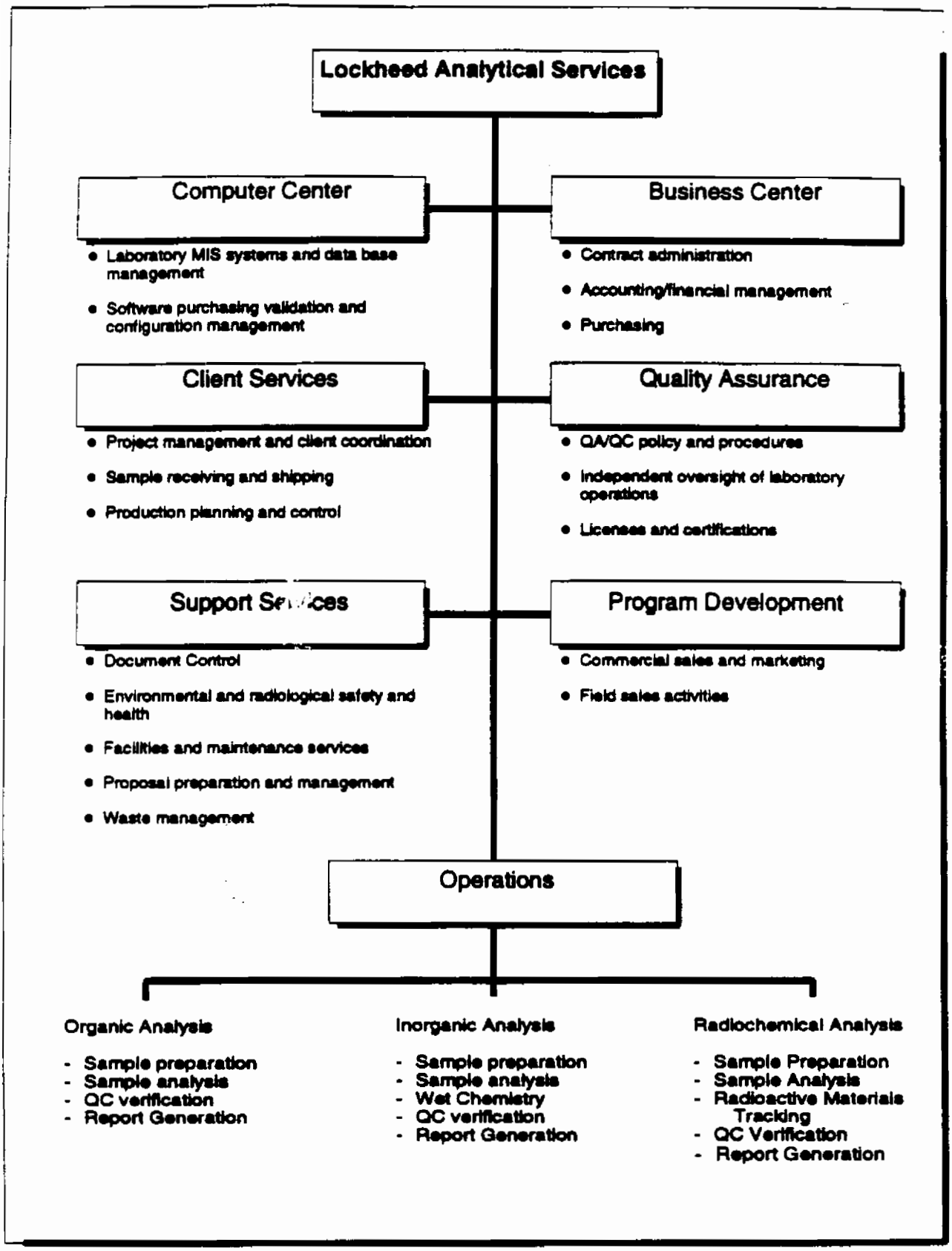
The LAS Director has the ultimate responsibility for ensuring that data and service quality meet or surpass the client's requirements. Additional responsibilities include:

- Supporting quality assurance as an essential requirement in all functional, management, and administrative areas.
- Providing the resources necessary to support an effective, ongoing, and comprehensive quality assurance program.
- Communicating management's commitment to quality assurance throughout the organization.
- Motivating all personnel to achieve increasing levels of technical competence and responsibility.
- Holding formal LAS Quality Assurance Program reviews to provide a forum for determining ways to improve LAS operations.

2.2.2 Quality Assurance Manager

The Quality Assurance Manager reports directly to the LAS Director and is responsible for designing, implementing and maintaining the LAS Quality Assurance Program in a timely, accurate, and consistent basis, and for taking or recommending corrective actions, as required. Additional responsibilities include:

Figure 2. The LAS Organizational Structure



- Establishing and maintaining a comprehensive, effective quality assurance program.

- Developing, evaluating, and documenting quality assurance policy and procedures appropriate to all laboratory functional areas with the LAS management.

- Ensuring that all laboratory operations are conducted in accordance with the LAS Quality Assurance Program and with the QA and QC requirements specific to each analytical method.

- Ensuring that all laboratory activities comply with local, state, and federal environmental regulations.

- Reviewing project-specific quality assurance plans.

- Ensuring that QC limits are established and followed for critical points in all measurement processes, and that they are based on sound statistical methods.

- Initiating internal performance audits using certified, high-purity standard reference materials (SRMs) purchased commercially.

- Performing independent QA review of a predetermined quantity of data reports.

- Informing management of system breakdowns or deficiencies, recommending corrective actions to improve the data-generation system, and defining the validity of data generated during all out-of-control situations.

- Preparing and revising quality assurance-related documents (e.g., SOPs) and periodic quality assurance reports to management.

- Advising and training laboratory staff in quality assurance practices central to their work.

- Conducting periodic technical systems evaluations of the laboratory facilities, instrumentation, and operations.

- Coordinating interlaboratory comparison studies.

- Overseeing the evaluation of locally developed computer software.

- Overseeing the LAS Training Program activities.

- Evaluating subcontractors and vendors that provide analytical and calibration services.

- Administering the laboratory accreditation and licensing activities.

2.2.3 Operations Manager

The Operations Manager reports directly to the LAS Director and is ultimately responsible for timely and accurate analysis of samples and generation of data. The Operations Manager responds to analytical requests for quotation and recommends proper analytical methods. Furthermore, this individual has the authority to reject samples that are inappropriate for analysis and to request repreparation and reanalysis of samples that are of questionable technical quality. The Operations Manager is responsible for overseeing the following laboratory functions:

- Status and progress of analytical workload.

- Coordination of sample preparation and analytical work.

- Analytical data production and review.
- Report production.

2.2.4 Client Services Manager

The Client Services Manager reports directly to the LAS Director and serves as the interface between the laboratory and the client. The Client Services Manager oversees the following functions:

- Working with clients to define requirements for analytical methodologies, quality assurance, report and deliverable timelines, and pricing.
- Working with LAS management to determine acceptable scheduling.
- Informing managers of the pending arrival of samples for analysis.
- Identifying and providing oversight of analytical services subcontractors and receipt of data reports.
- Tracking revenue/sample projections and reviewing invoices for completed work.
- Predicting potential workload.
- Sample receipt, log in, identification, storage, and tracking.
- Managing inventories of reagents, solvents, and materials.

2.2.5 Program Development Manager

The Program Development Manager reports directly to the LAS Director and oversees the following functions:

- Developing and implementing effective marketing and sales strategies.
- Identifying market trends, including new areas of service and revenue projections.

2.2.6 Support Services Manager

The Support Services Manager is responsible for providing administrative, maintenance, security, safety, and health support services for the timely implementation of laboratory operations. Specific responsibilities include overseeing the following activities:

- Maintenance and security of laboratory support equipment and facilities.
- Maintenance and archiving of laboratory records and documents.
- Developing programs to ensure compliance with laboratory environmental and radiation safety and health requirements.
- Providing waste management services.

2.2.7 Business Manager

The Business Manager oversees the functions of purchasing, accounts payable, payroll, property control, financial forecasting, invoicing, accounts receivable, and administers laboratory contracts.

2.2.8 Information Systems Manager

The Information Systems Manager reports directly to the LAS Director and oversees the following functions:

- Directing the laboratory data system (Oracle based) operations.

- Overseeing operation of network hardware and software, including distribution of personal computers (PCs).

- Developing, testing, and modifying new or existing data base applications.

- Controlling access to the laboratory minicomputer thorough management of user accounts.

- Providing technical support to LAS staff for problem solving and strategic planning.

2.2.9 Supervisors

Supervisors provide the critical link between upper management, laboratory technical personnel, and support staff. Section supervisors support the Operations Manager in formulating laboratory policy and in planning and scheduling laboratory operations. Supervisors oversee and direct the work flow in their respective groups on a daily basis. Areas of supervisory responsibility include:

- Scheduling sample preparation and analysis with regard to holding time, QA and QC, and data turnaround requirements.

- Offering guidance in the selection of equipment and methods.

- Providing guidance in resolving analytical and other technical problems encountered during sample and data preparation, analysis, and documentation.

- Assigning work priorities within the group.

- Keeping abreast of (1) preventive maintenance and repairs necessary for all equipment in the group and (2) inventory of consumables in stock.

- Ensuring that technical review of 100% of the data generated by the section is performed.

- Checking logbooks, data reporting forms, etc., on a scheduled basis.

- Reviewing completed project work.

- Ensuring that all new employees in the group receive proper training and are qualified and proficient in performing assigned tasks.

- Participating in staffing selection for the group.

Any or all of these functions may need to be delegated to technical specialists or other senior staff.

2.2.10 Laboratory Technical Personnel

The nonsupervisory technical staff of the laboratory must understand the importance of the LAS Quality Assurance Program and their individual responsibilities in ensuring the success of the program. The individual shall maintain continuous awareness of good laboratory and safety practices, recognize potential sources of error in assigned tasks, report observed substandard conditions or practices, and generally use good judgment in daily activities. In particular, each technical employee is responsible for:

- Implementing the policies contained in this document.

- Using QC procedures and SOPs properly during sample preparation, sample analysis, data generation, or any routinely performed activity.

- Properly maintaining complete documentation of laboratory activities.

- Correcting and thoroughly documenting problems and deficiencies in any portion of the measurement system.

- Evaluating 100% of data for acceptability, on the basis of method-specified QC limits and professional judgment.

- Ensuring that all laboratory operations are performed according to the appropriate governmental and laboratory health and safety program.

2.3 TRAINING PROGRAM

Training ensures (1) that the LAS staff consistently produces high-quality work; (2) the individual, regardless of his or her technical, managerial, or support role, is capable of performing the assigned task, (3) has the proper level of education and background for the specific job classification, (4) has received the appropriate orientation and indoctrination into LAS operations; and (5) proper mechanisms are in place to enhance employee skill levels. The quality assurance aspects of job performance are central to the training that LAS technical personnel receive. The LAL-91-SOP-0110 provides a detail description of the training program requirements. The following subsections provide an overview of the training program at LAS.

2.3.1 Orientation and Indoctrination

Orientation introduces the individual to existing systems and operating conditions, and indoctrination trains the individual in the principles and operations of those systems and tasks. LAS Quality Assurance Department representatives and laboratory supervisors train staff and instruct them in task-specific QC data recording and related activities. In general, in addition to instruction in environmental safety and health aspects of their positions, LAS technical staff are trained in the following areas as they apply to their positions:

- Purpose and significance of the LAS Quality Assurance Program.

- Standard operating procedures.

- LAS Quality Assurance Policy and ethics related to analytical data production.

- Overview of federal, state, and local regulations.

- LAS Notebook Policy and Guidelines for Maintaining Laboratory Logbooks and Records.

- QA and QC responsibilities.

- Nonconformance and corrective action identification and documentation.

- Client-specific data reporting requirements.

- Proper use and frequency of blanks, replicates, spikes, surrogates, and other QC samples and implementation of corrective actions.

- Technical review of analytical data.

- Standard reference material traceability.

- Proper receipt and handling of environmental samples (chain of custody).

- Data archival and retrieval practices.

Furthermore, the Operations Manager, the section supervisors and the technical staff are responsible for providing continuous, on-the-job

training by monitoring activities of subordinates and by preparing and reviewing SOPs. In some instances, individual training may be appropriate so that the new employee will learn correctly and rapidly. This approach provides knowledge and the technical skills that are necessary to perform the required tasks and it emphasizes the importance of high-quality performance. Specifically, each LAS technical employee shall be trained in the following basic laboratory operations as they relate to his/her task assignments:

- Sample receipt and logging.
- Sample handling and measuring.
- Instrument tuning and calibration.
- Data recording, handling, and reporting.
- Support equipment maintenance.
- Sample tracking through LDMS.
- Waste handling.

Dry runs and simulations of sample processing activities may be incorporated into the training program to ensure that protocols are clearly understood and that problems are identified and solved before a project begins.

2.3.2 Proficiency Monitoring and Retraining

Personnel who must perform tasks that require special skills or abilities must first demonstrate their proficiency in the assigned task under an instructor's supervision. Proficiency of each analyst is monitored by the section supervisor or designee by reviewing the analytical data generated from repetitive preparation and measurements of QC samples such as laboratory control standards, matrix spikes and matrix spike duplicates, replicates, surrogate spikes, method blanks, and blind PE samples. Independent blind PE materials obtained from approved vendors are routinely submitted by the QA Department staff to the operations staff to assess and monitor instrument, method, and analysts' performance in analyzing inorganic and organic constituents in various sample matrices. Results are evaluated against the vendor's certified acceptance limits. Any analysis that is associated with unacceptable results is further monitored by sending another set of PE materials. An overall passing score of 80 percent based on the number of acceptable analyte determinations in the PE sample is used to assess performance level. On the basis of unacceptable results, the QA staff may recommend that the sample preparation staff or the analyst need additional training. The proficiency of analysts is also monitored through successful analysis of external PE studies. The result provides a practical demonstration of the ability of the analyst to maintain performance within acceptance limits. This approach also provides a way to identify deficiencies in performance, to learn more about a new method, to understand a new project, and to learn how to use an instrument and solve problems. Qualification is considered valid for approximately two years (unless revoked for a reason) at which time the person's qualifications will need to be reevaluated and requalified.

Furthermore, to provide safe working conditions and to protect the public health, all new LAS employees who may be exposed to hazardous materials receive training from the LAS Safety Officer before they begin work. The Safety Officer schedules and presents hazardous materials awareness, hazardous materials Phase I, hazardous materials Phase II, and radiation safety courses. Training on laboratory safety is given when an employee is assigned new duties.

2.3.3 Training Records

This vital activity is thoroughly documented in the form of technical, SOP, and proficiency training records; need assessment for procedure specific training; training attendance sheets; SOP review records; personnel training records; hazard communication standard documentation; and memoranda describing the specific training activities.

Qualifications of all professional, technical, and support personnel are documented via resumes, which include academic credentials, employment history, experience, and professional registration or certification, as appropriate for the position.

A data base provides essential information on the current status of the procedure-specific training that each technical staff member has received.

Copies of all training records are maintained in a secure file cabinet. QA staff members coordinate the entry and filing of the appropriate documents to the training data base and training files.

CHAPTER 3
DATA QUALITY OBJECTIVES

Data Quality Objectives (DQOs) are the foundation for collecting environmental data that can provide a reliable basis for decisions concerning environmental remediation. The quantitative measurements that estimate the true value or true concentration of a physical or chemical property always involve some level of uncertainty. The uncertainty associated with a sample generally results from (1) natural variability of the sample, (2) sample handling operations and conditions, (3) spatial and temporal patterns, and (4) analytical variability. For an environmental data collection project, the uncertainties must be estimated and compared to standard, quantitative indicators of data quality (i.e., DQOs).

Typically, DQOs are identified during project scoping and development of sampling and analysis plans. In this manual, however, we discuss only the analytical DQOs because LAS generally does not have any jurisdiction over sample collection, shipment, or other field-related activities that may affect the data quality of the environmental sample before the sample is received in the laboratory.

DQOs are established to meet method- or client-specific requirements and to ensure that the data collected are sufficient and of adequate quality for their intended uses (EPA, 1987a). EPA has established six primary analytical DQOs for environmental studies: precision, accuracy, representativeness, completeness, comparability, and detectability. Section 3.1 addresses precision and accuracy which is augmented by Appendix A which lists analytical DQOs for precision, accuracy, and detectability for inorganic, organic, and radionuclide constituents. Sections 3.2 through 3.5 address the qualitative requirements for completeness, representativeness, and comparability, and detectability, respectively.

The components of analytical variability (uncertainty) can be estimated when QA and QC samples of the right types and quantities are incorporated into measurement procedures at the analytical laboratory. At the LAS, numerous QA and QC samples are analyzed to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The QA and QC samples and their applications, described in tables 2 and 3, are selected on the basis of method- or client-specific requirements. Field blanks, field duplicates, and performance evaluation (PE) samples, which are described in Table 2, are received from the client as unknown samples. Analytical laboratory QC samples for inorganic, organic, and radionuclide analyses include calibration or instrument blanks, method blanks, background, duplicates, replicates, laboratory control samples (LCSs), calibration standards, matrix spikes (MSs), matrix spike duplicates (MSDs), surrogate spikes, and yield tracers.

3.1 PRECISION AND ACCURACY

Precision is an estimate of variability. In other words, it is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions (EPA, 1980). The precision of a measurement system is affected by random errors. Precision is expressed either as relative standard deviation (RSD) for replicate measurements greater than two or as relative percent difference (RPD) for duplicate measurements. RSD for replicate measurements ($n > 2$) is calculated as a percentage:

Table 2. Descriptions and Applications of QA Samples

Sample Type	Type of Analysis*	Description	Application
Performance evaluation (audit) samples	I, O, R	Homogeneous, stable, and certified synthetic audit sample	Estimate intra- and inter-laboratory bias and estimate system precision
Field blank	I, O, R	ASTM Type II water that is carried through the same system as the field samples	Identify contamination resulting from sampling operations and estimate system detection limit
Field duplicate	I, O, R	Second sample collected at the sampling site	Estimate system precision

* = I - Inorganic analysis, O - Organic analysis, R - Radionuclide analysis.

$$\%RSD = \frac{s}{\bar{X}} \times 100$$

where s is the standard deviation of the series of individual measurements and $\bar{X}$ is the mean of the series of individual measurements.

The mean of the set of individual measurements is determined by summing the values for the individual measurements ($X_1 \ldots X_n$), then dividing by the number of measurements (n):

$$\bar{X} = \frac{X_1 + X_2 + X_3 + \ldots + X_n}{n}$$

The standard deviation for this set of measurements then is determined as follows:

$$s = \sqrt{\frac{\sum (X_i - \bar{X})^2}{n - 1}}$$

where X_i represents each of the individual measurements.

The RPD is calculated in percent for duplicate measurements as follows:

$$\%RPD = \frac{X_1 - X_2}{(X_1 + X_2) / 2} \times 100$$

where X_1 is the first sample result and X_2 is the duplicate sample result.

For radionuclide determinations, replicate measurements must agree within the 95 percent confidence level based on the summed error of the analysis, or within 3σ of the weighted average, as required by the client-specific protocol and is measured by the replicate error ratio (RER).

The RER or duplicate error ration (DER) for radionuclide determination is calculated as follows:

$$RER = DER = \frac{R_1 - R_2}{(\sigma_1 + \sigma_2)}$$

Where:

R = Result
σ = 95% confidence level

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value

Table 3. Descriptions and Applications of QC Samples

Sample Type	Type of Analysis[a]	Description	Application
QC Check Standard (ICV, CCV, QCCS)	I,O	Independent standard; prepared from source other than calibration standard	Indicates accuracy and consistency of calibration
Laboratory control sample	I,O,R	Independent standard; processed through the entire analytical procedure	Indicates accuracy of the analytical procedure
Detection limit QC check sample	I,O	Standard at a constituent concentration 2 to 3 times the detection limit	Indicates accuracy at lower end of calibration range
Check Source	R	Pure standard containing radionuclides at specified levels	Provides a check on counting instruments
Calibration blank	I,O	ASTM Type II water or better (zero constituent concentration)	Indicates instrument signal drift and sample contamination
Method blank	I,O,R	All reagents used during sample preparation steps (i.e., digestion, extraction, distillation)	Indicates sample contamination introduced during sample preparation steps
Background	R	Instrument background (e.g., deep well water for tritium)	Indicates instrument background level
Internal standard	O	Constituents added to every sample at a known concentration; not expected to be detected in environmental media	Measures the relative responses of other method constituents and surrogates
Surrogate spike	O	Constituents not expected to be detected in environmental media; added to every sample at a known concentration	Evaluates sample preparation and analytical efficiency
Quench source	R	Standard that contains quenching materials for liquid scintillation counting	Indicates liquid scintillation counting efficiency
Analytical laboratory duplicate/replicate	I,R	Sample aliquot; split at the analytical laboratory	Indicates analytical precision
Matrix spike	I,O,R	Sample plus known quantity of constituent	Indicates sample matrix effect on analysis and on accuracy of measurement system
Matrix spike duplicate	O,R	A second matrix spike sample	Indicates analytical precision influenced by sample matrix
Yield tracer	R	A radioactive or nonradioactive isotope at a known, specified quantity	Traces yields during sample preparation

[a] = I - Inorganic analysis, O - Organic analysis, R - Radionuclide analysis

(EPA, 1980). Systematic errors affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100):

$$\%R = \frac{X}{T} \times 100$$

where X is the measured standard value and T is the true or expected (reference) value.

The precision and accuracy DQOs that are to be used in evaluating inorganic, organic, and radionuclide constituents at LAS are provided in method-specific SOPs and in the documentation for the analytical method of interest.

Precision and accuracy are determined, in part, by analyzing data from matrix spike and matrix spike duplicates, unspiked duplicates, LCSs, and single blind audit samples (see tables 2 and 3). For radiochemical determinations, counting statistics can also provide an estimate of uncertainty.

3.2 COMPLETENESS

Completeness is a measure of the percentage of measurements that are judged to be valid measurements (EPA, 1980). At a minimum, the objective for completeness of data is 90% for each constituent analyzed.

3.3 REPRESENTATIVENESS

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition (EPA, 1980). Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result (concentration) is representative of the constituent concentration in the sample matrix. The LAS analysts will make every effort to analyze an aliquot that is representative of the original sample, and the homogeneity of the sample will be ensured before subsampling.

3.4 COMPARABILITY

Comparability is a measure of the confidence with which one data set can be compared to another (EPA, 1980). The comparability standards for LAS require that all laboratory analysts use uniform procedures and a uniform set of units and calculations for analyzing and reporting environmental data. To ensure that employees clearly understand the requirements and standard protocols, all personnel will participate in the uniform, ongoing LAS training program.

3.5 DETECTABILITY

Detectability refers to the minimum concentration of a constituent that can be measured by a measurement system with a stated level of confidence (Taylor, 1987). It is determined by assessing the variability of replicate measurements at zero or near zero constituent concentration, and it is reported in concentration units.

It is LAS policy to determine, for each inorganic and organic constituent, the instrument detection limit (IDL) or method detection limit (MDL) and the reporting detection limit (RDL) before any samples are analyzed. For radionuclide constituents, minimum detectable activity (MDA) is determined.

3.5.1 Instrument and Method Detection Limits

In general, a detection limit is the point or concentration at which a measured value becomes believable. In other words, it is the point at which the value is larger than the uncertainty (e.g., noise level) associated with it. The detection limit is defined as the smallest observed signal with the reliability of 1 minus α (where α is the probability of Type I error) that can be considered a signal caused by the constituent of interest.

For inorganic and organic constituents, detection limits are estimated by determining the standard deviation (s) from the results of the 7 to 10 measurements of the low-level standards or a blank analyzed on the same day. The IDL and MDL differ, not in how they are calculated, but in the way in which the low-concentration standards are handled before analysis. Because MDLs are sensitive to instrument and matrix effects, the MDL is determined by allowing the standard to undergo the appropriate sample preparation technique (i.e., extraction, digestion, distillation), as required by the analytical method before analysis. On the other hand, the IDL is calculated from standards that are not subjected to these handling steps. Thus, aside from sample preparation, all procedures for determining the IDL and MDL are the same. The distinction is that the IDL estimates the detection limit of the instrument under ideal conditions, whereas the MDL estimates the detection limit in more practical terms.

For each method and each analyte required by the method, the instrument detection limits (IDLs) are determined on a quarterly basis for metals constituents and on an annual basis for other inorganic constituents and method detection limits (MDLs) are determined annually for organic analyses.

The IDL or MDL is computed as three times the standard deviation (i.e., $3s$) of replicate (7 to 10) runs of a standard in which the concentration of the analyte of interest is at or near the detection limit specified for that technique. The replicate measurements of the low-level standards are performed on three nonconsecutive days for inorganic analyses and on the same day for organic analyses. The concentration of the standards needs to be at approximately three to five times the expected or method-specified detection limit.

The MDL studies are intended to estimate the lowest concentration of an analyte that can be routinely determined by a method with less than a 1% probability of getting a false positive measurement. The procedure involves performing multiple measurements of the analytes at a concentration that is near the expected MDL. A Student's t test is performed on the multiple measurements, and the MDL is set at the value where the measurements are distinguishable from zero at the 99% confidence level.

The variability in an analytical measurement is assumed to consist of two distinct portions. One portion is proportional to the concentration of the analyte in the sample. Small errors in volumetric measurements and transfers will create errors that are proportional to the initial analyte concentration. Since the MDL studies are run at a very low concentration, this component of the error is assumed to be low. The other portion of the variability is due to the measurement noise and chemical interferences. The objective of the MDL study is to estimate this second type of error while minimizing the first type.

3.5.2 Reporting Detection Limits

The RDL is the lowest level at which measurements become quantitatively meaningful

The RDL is defined as approximately 3 times the MDL. Because sample-handling activities, in addition to sample analysis, have an influence on detecting the presence or absence of a constituent, obtaining extremely low IDLs in the laboratory is meaningless in relation to the environmental sample result. Therefore, for reporting data, the LAS has adopted the approach of determining RDLs, which include the variability that may result from sample preparation, sample handling, and analysis techniques. For each constituent and each instrument used to quantitate that constituent, the experimentally determined IDLs and MDLs must be less than the RDLs shown in Appendix A.

The RDL is established to account for a number of factors. First, greater relative error is expected for analyte concentration at or near the IDL or MDL which decreases the level of confidence in the accuracy of the analyte quantification. Second, there is some day-to-day variability in both the IDL or MDL which means that on a given day it may not be possible to detect an analyte at the statistically determined MDL. Third, environmental samples are rarely as clean or as analytically straightforward as the matrices used to determine detection limits. Consequently, the RDL is set above the MDL and IDL (1) to reduce relative error, (2) to account for expected variability, (3) to ensure that analytes can be reported at the RDL with reasonable level confidence, and (4) to reduce the chance of reporting false negatives in environmental samples.

Two final factors in establishing RDL values include (1) the ease of data interpretation for our clients and (2) standardization of data reporting for the laboratory. If the RDL values were to change each time a new IDL or MDL study was performed, this would cause confusion to our clients and would be difficult to maintain consistency in reporting for the laboratory staff, which may result in reporting of erroneous data. Therefore, the RDL is established at a level high enough so that changes to these values are infrequent.

This is the set of statistical assumptions behind the detection limit studies. The assumptions, however, are not always correct. The results from the detection limit studies should not be taken at face value; the results should be scrutinized closely and adjusted based on the experience of the scientists who are involved in performing the study. For example, some MDL studies may show very little variability for constituents that have historically been difficult to detect. In these cases, the MDL is probably unrealistically low, and it should be adjusted upward based on the experience of the analyst and the QA specialist. In other cases, variability in the sample preparation or other factors may result in obtaining a high MDL, while observation of the raw instrument output indicates that there is adequate instrumental sensitivity to detect the analyte at concentrations far below the measured MDL. In these cases, the MDL should be lowered to reflect the true state of the instrument performance.

3.5.3 Minimum Detectable Activity

The minimum detectable activity (MDA) is a measure of the quantity of radioactive materials that could be present and detected by the analysis. The MDA is reported in activity units per unit volume or weight, such as pCi per liter or pCi per gram. Many factors affect the MDA, including calibration geometry, backgrounds (system and source-induced), detector resolution, counting systems, sample sizes, and the particular isotope measured. With the exception of the MDA formula chosen. the MDA is not affected by the analysi software. It is the LAS policy to calculate MDAs as described in ANSI 13.30 and NRC

Reg guide 86-16 also known as the "Currie Method" unless otherwise specified by the client.

By using state-of-the-art instrumentation, (e.g., a high-resolution gamma spectrometer with a 96% efficiency detector, an alpha/beta counter with less than 0.1 counts per minute [cpm] background alpha and less than 1 cpm beta, or a liquid scintillation counter with tritium channel having less than 0.8 cpm background) lower MDAs can be achieved with less counting time than required by using conventional instrumentation.

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CHAPTER 4
SAMPLE MANAGEMENT

4.1 INTRODUCTION

Sample management is the overall process by which samples are controlled, transferred, handled, and stored from the time of collection through analysis and final disposition. Sample management and chain of custody are closely related. Sample management refers to those activities aimed at ensuring sample integrity. Chain of custody involves establishing accountability for the sample; documenting how the sample is received, tracked, and stored; and defining who has access to and handles the sample. Internal chain-of-custody procedure is addressed in Chapter 13 of this plan and in LAL-90-SOP-0009, and sample receiving and log in are described in LAL-90-SOP-0002.

Sample management begins in the field when the sample is collected. The management of the collection process, with the exception of providing sample containers, is not within the jurisdiction of the LAS and is not discussed in this plan. All other sample management activities, specifically those related to sample shipment, sample containers, sample preservation, sample holding times, and sample preparation and analysis, are discussed here.

4.2 SAMPLE AND LABORATORY CONTAINERS

Container specifications depend on the client requirements, in addition to analytical method, constituent, and sample matrix of interest. LAS purchases commercially precleaned bottles for which the supplier has certified in writing that the cleaning procedure used meets or exceeds all CLP container guidelines (Chapter 16). This specification applies to all shipping and sample containers and all reagents, whether shipped off site or used only at LAS.

In general, use the following containers:

- 40-mL Teflon®-septa glass vials (clear or amber) for aqueous samples to be analyzed for volatile organic constituents (designated as "Level I").
- 1,000-mL amber glass bottles with Teflon®-lined caps for aqueous samples to be analyzed for semivolatiles, pesticides, and PCBs (designated as "Level I").
- Polyethylene bottles for aqueous samples to be analyzed for inorganic constituents (designated as "Level I").
- Wide-mouth, amber glass bottles for all soil and sediment samples.

All containers must be kept in a contaminant-free, secure area. Detailed descriptions of container specifications are given in Appendix B. Sample containers shall not be cleaned and reused for any other analyses. LAL-90-SOP-0045 provides guidelines for sample container types and uses.

In addition, use the following guidelines when selecting the material composition of sample containers and laboratory vessels:

- Use borosilicate or polyethylene bottles for storage of reagents and standard solutions, unless otherwise specified; however, do not use plastic containers for reagents and standard solutions used in organic analyses.

- Borosilicate glassware is not completely inert, especially to alkali materials. Store standard solutions of silica, boron, and the alkali metals in polyethylene bottles.

- Dilute metal standard solutions have a tendency to "plate out" on container walls over time; therefore, prepare these solutions immediately before analysis.

- Disposable glassware is acceptable for some analyses. For example, disposable vials and cuvettes are appropriate for use in automatic samplers. However, every effort must be made to ensure that these containers are free of contamination (i.e., target constituents or interferants) that can affect the analytical results.

4.3 SAMPLE PRESERVATION

Sample preservation prevents or retards the degradation and/or reaction of chemicals or biological activity in samples during transit and storage. Efforts to preserve the integrity of the samples are generally initiated at the time of sampling and should continue until analyses are performed. Preservatives are typically added to the sample container at the time of sample collection. (Note: Preservatives are added to aqueous samples only.) However, if requested by the client, LAS will send to the field pre-measured volumes of the preservatives in sealed ampules or in sample containers. Since pre-measured volumes of preservatives added at LAS may not always be sufficient to preserve samples, samplers are ultimately responsible for ensuring adequate preservation. Preservation and storage guidelines and requirements, by constituent, are provided in Appendix B.

4.4 SAMPLE HOLDING TIMES

The maximum time that a preserved sample may be held between sample collection and analysis depends on the stability of the constituents of interest. Holding-time limitations are intended to minimize chemical changes in a sample before it is analyzed. Maximum allowable holding times provided in Appendix B apply to aqueous, soil, and sediment samples when proper preservation procedure is followed. Holding times are calculated from sample collection to analysis, unless otherwise specified by the method.

It is LAS policy to adhere to the method or client-specified maximum holding time requirements. To expedite analysis and to minimize the possibility of exceeding holding times, it is imperative that samples be sent to the analytical laboratory by a fast, reliable method as soon as they are collected. Following sample receipt and log in, the appropriate section supervisor schedules the analysis of samples to ensure that all samples are analyzed within holding times. Holding times for sample preparation (i.e., extraction, digestion, or filtration) and analysis are entered into the sample receipt data base to ensure that samples are prepared and analyzed within the prescribed holding-time for each constituent of interest.

For some constituents, it may be necessary to hold samples in cold storage even if holding times have been exceeded. The extended storage is necessary to ensure that the samples are suitable for possible reanalysis. If the original analysis of the sample or any subsequent reanalyses exceed the holding times

for the particular constituents, a statement must be included in the case narrative of the analytical data report, and affected sample results must be qualified appropriately to indicate that the holding time requirement has been exceeded.

4.5 SAMPLE PREPARATION

The matrix of the sample and the constituents to be analyzed affect the selection of sample preparation steps. Sample preparation procedures for organic, inorganic, and radionuclide analyses are provided in various method-specific SOPs (see Appendix C).

Water used in the course of inorganic, organic, and radionuclide analyses (dilutions, preparations of standard and blank samples, etc.) must meet or exceed the standards for purity of ASTM Type II grade reagent water. All extracts, digestates, and filtered samples are collected in specified sample containers and are labeled and tracked on chain-of-custody forms and in the respective laboratory logbooks. Samples and final sample preparations (e.g., organic extracts) are stored at 4°C (± 2°C), except for metals digestates, which are stored at room temperature in acidic solutions resulting from the digestion process. Samples and standards shall be stored separately. Samples designated for VOC analyses are maintained in designated refrigerators separate from samples for other organic analyses to prevent cross contamination. The Quality Assurance staff monitors refrigerators designated for VOAs for potential cross contamination by using holding (refrigerator) blanks.

For most inorganic analyses, chemical reagents, solvents, and gases of the analytical reagent grade are used. For other analyses, such as trace organic and radiochemical, special ultrapure reagents, pesticide-quality and analytical-grade solvents, and gases are used as required for the method of interest. For methods in which the purity of reagents is not specified, LAS uses analytical grade reagents, solvents, and gases. A detailed discussion on the reagent requirements is also provided in Chapter 16.

4.5.1 Subsampling for Sample Preparation

Many environmental samples are heterogeneous upon receipt and may require some physical manipulation in order to yield representative (i.e., homogenous) sub-samples. LAS-91-SOP-0105 provides procedures for subsampling liquid, semisolid, and solid samples, and samples containing inorganic and organic constituents.

4.5.2 Filtration

The need to filter aqueous samples depends on whether total or dissolved constituents are of interest. Samples to be analyzed for dissolved inorganic constituents must be filtered in the field if preservation is needed. Samples for dissolved metals analyses must be filtered in the field before chemical preservatives are added to preclude the release of contaminants from the particulate matter. However, if the client requires that the samples be filtered in the laboratory under a controlled environment, the filter material used must be compatible with the constituents of interest.

4.5.3 Extraction and Digestion

Many organic (e.g., semivolatile, pesticides) analyses of environmental multimedia samples require the extraction of sample constituents of interest into organic solvents before analysis. Many metals analyses of aqueous samples require digestion of the sample. SOPs delineating these preparation steps are listed in Appendix C.

Soils and sediments are complex mixtures of widely varying compositions, even within a single site. Recovery of constituents depends on many factors, including organic content, mineral content, particulate size, and moisture content of the soil. It is LAS policy to report soil and sediment sample analyses for organic and inorganic methods in the as-received condition (i.e., wet weight) unless the client specifies that dry weight is to be reported. For radiochemical determinations, data are routinely reported based on dry weight of the sample.

For radionuclide determinations, grinding of samples may be necessary to ensure the collection of homogeneous representative sub-samples.

4.6 SAMPLE ANALYSIS

All samples shall be processed through the entire analytical method, as specified in Chapter 5. All analyses shall be performed within the appropriate calibration range (Chapters 6, 7, and 8) of the instrument. Each sample for which the constituent concentration exceeds the calibration range shall be diluted and analyzed within the appropriate analytical range. Records of all dilutions shall be maintained in analysis or injection logbooks, and dilution factors shall be reported on the appropriate data reporting form. The method of constituent identification and quantitation is specified in the analytical methods.

4.7 SAMPLE TRANSFER BETWEEN PREPARATION AND ANALYSIS PERSONNEL

LAL-90-SOP-0002 and LAL-90-SOP-0009 describe the procedures for sample handling and internal tracking pertaining to the original sample and the sub-samples. All analysts are trained in the proper tracking and storage of samples.

In summary, the sample is initially transferred from the sample custodian to the sample (organic, inorganic, or radiochemistry) preparation personnel who take custody of samples. For cases in which there is no sample preparation (e.g., volatiles, anions), custody is transferred directly to the analyst. The extracted, digested, or filtered portion of the sample used for analysis remains in the preparation laboratory under specified storage conditions until analysis is scheduled and the unused, raw portion of the sample is returned to the sample custodian for proper storage in the prescribed area. If reanalysis is required on the raw sample, the sample is obtained through the sample custodian, and standard chain-of-custody and sample-tracking procedures are followed.

Within the inorganic suite, there are two discernable areas: one that is refrigerated and one that is maintained at ambient temperature. Samples that are not returned to the sample log-in area after use are stored in these areas to further ensure the integrity of the environmental samples. The tracking sheets used in the laboratory and the LDMS indicate where samples are stored while they are being processed in the inorganic suite.

4.8 SAMPLE DISPOSAL

There are several possible ways to dispose of the sample after use:

- The sample will be disposed of as hazardous waste; or
- The sample may be returned to the client, if requested.

It is LAS policy to dispose of samples 60 days after the submittal of the report to the client unless otherwise specified in writing by the client or as required by regulations and licenses governing laboratory operations. However,

samples will not be disposed of until contract-required terms are expired or until the client is notified in writing of the intent to dispose. Complete records of the disposal date and method will be maintained along with all the other sample-tracking information maintained in the Document Control files. Waste disposal follows RCRA and other applicable protocols. LAL-90-SOP-0003 provides a detailed discussion of general LAS waste-handling considerations. LAL-91-SOP-0083 addresses disposal of radioactive materials.

4.9 PREVENTION OF SAMPLE CONTAMINATION IN THE LABORATORY

To ensure that sample integrity is maintained throughout the laboratory, LAS staff must follow GLPs and GMPs in the handling, preparation, and analysis of environmental samples. Chemical, physical, and radionuclide determinations must be performed in a work environment free of sample contaminants and free of constituent and measurement interferences. LAL-90-SOP-0004 addresses the specific methods to prevent potential sample contamination.

Special precautions must be taken during sample handling and analysis to minimize or eliminate cross contamination because environmental samples sent to LAS for analysis often contain trace concentrations of constituents. In general, all work areas must be kept clean and free of dust and dirt accumulation. All counter tops and chemical fume hoods where sample preparations and wet chemical analyses are performed must be cleaned regularly.

4.9.1 LAS Facility Features

Special structural design features at LAS minimize or prevent sample contamination that may occur during sample handling. The significant design features include:

- Segregated volatile and semivolatile organic analysis laboratories to minimize potential sample contamination associated with the use of common organic solvents such as methylene chloride, hexane, and acetone.

- Separate glassware washing facility supplied with high purity reagent water.

- Air balancing systems that (1) change the air 10 times per hour to help reduce the presence of airborne contamination primarily for commonly used organic solvents, (2) maintain positive pressure in the volatile organic analysis laboratory relative to the hallway to ensure that airborne contaminants do not enter the laboratory, and (3) maintain negative pressure in the other laboratories relative to the hallways to prevent airborne contaminants from escaping into the rest of the building.

- 36 chemical fume hoods, 18 snorkels, and 2 perchloric acid hoods to carry off fumes and to reduce the risk of aerosol and airborne contaminants and of personal safety hazards in the laboratory.

- Exhaust stacks from the hoods in the radioactive section that pass the exhaust from the hoods through scrubbers or HEPA filters to prevent the release of radioactive contaminants into the environment.

4.9.2 Reagents and Reagent Water

The quality of the chemical reagents, solvents, and gases used for organic and inorganic analyses must meet minimum requirements for analytical reagent grade or requirements

specified by the particular method (see Chapter 16 for details). Standards, reagents, and solvents must be stored according to manufacturer's guidelines. Photosensitive reagents and standards are stored in appropriate dark storage areas.

Laboratory reagent water used for dilutions, preparation of standards and blanks, and glassware cleaning is generated using the Barnstead/Thermolyne NANOpure Ultrapure Water System (Model D4741) in combination with dechlorination R.O. and mixed-bed ion exchange. This system is designed to supply water that meets requirements for ASTM Type II reagent water or better.

4.9.3 Sample Storage

When required, samples are stored at 4 ± 2°C to preserve their integrity, as required by the analytical method of interest. Storage includes procedures that maintain the constituent levels (through the use of chemical preservatives) and physical maintenance of the sample (through temperature or light control). When storing samples in refrigerators or light-protected enclosures, there is a risk of cross contamination. Measures are employed to prevent cross contamination, such as segregating samples to be analyzed for VOCs from those to be analyzed for semivolatile compounds, segregating environmental samples from standards (particularly for VOCs), and preparing and analyzing holding blanks to monitor cross contamination.

4.9.4 Glassware Cleaning

All glassware used in sample preparation must be cleaned according to the procedures specified for the particular analysis method. LAL-90-SOP-0017 provides protocols for glassware cleaning for organic constituents, and LAL-90-SOP-0018 provides protocols for glassware cleaning for inorganic constituents. LAL-91-SOP-0172 provides protocols for glassware cleaning for radionuclides. In general, water-soluble substances can be washed out with hot or cold water and the vessel can be rinsed with deionized water. Other substances more difficult to remove may require the use of a detergent, organic solvent, persulfate cleaning solution, nitric acid, or aqua regia. Regardless of the method of cleaning, it is good practice to rinse "dirty" glassware with the last solvent used as soon as possible after use because material allowed to dry onto glassware is much more difficult to remove.

4.9.5 Assessment of Sample Contamination Levels

Potential for background contamination that may result from the sample containers, reagent water, reagents, and solvents used in extractions and digestions; cross contamination during sample storage; or chemical and physical interference or constituent carryover during analytical operations are evaluated on the basis of QA and QC data derived from instrument, method, and holding blank samples. If blank sample data exceed method-specific or LAS-established acceptance limits, the problem is investigated and resolved. As necessary, samples associated with out-of-control situations are reprepared and/or reanalyzed.

CHAPTER 5
ANALYTICAL METHODS

5.1 ANALYTICAL METHODS SELECTION

Methods used in sample preparation or analysis are selected to meet the specific needs and requirements of the client. LAS employs standard, officially approved (e.g., EPA, ASTM, APHA) analytical methods to quantify inorganic, organic, and radionuclide constituents in environmental media (e.g., water, soil, sediment, sludge). The examples of approved methods are given in Appendix A for the inorganic, organic, and radionuclide constituents. A detailed description of these procedures is provided in EPA SW-846 (September 1986 and revisions), EPA/600/4-79/020 (EPA, 1983), and ASTM Standard Methods (17th and 18th Editions). EPA CLP methods are provided in the most recent Statements of Work or earlier versions based on clients' requirements.

5.2 ANALYTICAL QUALITY CONTROL

Rigorous QA and QC procedures are incorporated into sample preparation and analysis activities. Internal QC checks on the analytical procedures are discussed in detail for inorganic constituents in Chapter 6, organic constituents in Chapter 7, and radionuclides in Chapter 8.

In general, before an analytical run, the analyst checks schedules and records for maintenance and calibration to ensure that all necessary tasks are current. An initial or continuing calibration check verification is then completed, and the difference between analyzed values and the known standards is calculated. If any calibration check sample exceeds the control limit of the method (see method-specific SOPs as listed in Appendix C), adjustments are made and the instrument is recalibrated, as appropriate. During the course of the analytical run, the analyst incorporates all applicable QC samples in accordance with method-specific SOPs. Following each QC sample analysis, the analyst performs the necessary calculations either manually or by using appropriate software. If any QC sample falls outside method-specific control limits, the problem is investigated and resolved, and corrective action is performed in accordance with the method, the SOP, or the project-specific QA Plan. All information related to the analytical run is documented in the injection and analysis logbooks. All calculations related to QC sample analysis and the types and frequency of QA and QC samples (e.g., audits, blanks, spikes) are described in detail in the method-specific SOPs for inorganic, organic, and radioanalytical analyses.

5.3 LAS STANDARD OPERATING PROCEDURES

To ensure and document that each operational system and analytical procedure is performed in a uniform, standard way, LAS has documented a series of SOPs. A complete list of LAS SOPs, many of which are referenced in this plan, is provided in Appendix C. In general, analytical SOPs follow EPA or other approved methods. All personnel must fully understand the specific SOPs pertaining to their duties. It is the section supervisor's responsibility to ensure that each employee has read and understood all appropriate SOPs related to task responsibilities.

Each SOP is approved and signed by the section supervisor/designee, Quality Assurance staff member, Health and Safety Officer, and the LAS Director. At times, changes in SOPs are required as a result of new instrumentation, methods, client-tailored needs, or improved procedures. Any change in an SOP requires the approval of the section supervisor and the Quality Assurance Manager or designee which is documented using the SOP Change Form. On an annual basis, as required by the formal review process, SOPs are reviewed to determine if SOP reflects current operations and are approved by the individuals mentioned above.

Work instructions also provide a mechanism to document simple instructions to the laboratory staff responsible for processing and analyzing samples and in generating reports. These instructions are equivalent to internal memorandums that are also used to disseminate project-specific information. They serve as quick references to items such as specific spiking constituents, spiking levels, calculations, sample preparation techniques, project-specific information, etc. Work instructions are approved by the section supervisor, Quality Assurance Manager, project manager, or by an appropriate designee.

5.4 CONTROL CHARTS

An essential element of the QA process is the ability to detect changes in analytical performance quickly. The control chart is an effective tool for this assessment because it records in real time the accuracy (bias) and precision of the appropriate parts of the measurement process. In other words, the control chart demonstrates statistical control.

At LAS, Shewhart charts using a single measurement (X chart) of the laboratory control samples (LCSs) are generated using EXCEL to monitor analytical data quality in terms of accuracy and precision. The Shewhart chart has a center line that is defined by the process or calculated by using the data to prepare the chart. The interval between the center line and the control limit is based on the assumption that data are normally distributed on the sample size of the subgroup and on the estimated standard deviation. Using these charts, the percent recovery of the control analyte in the LCS is plotted against the control limits sequentially as a function of time. Two types of limits are established to judge whether a data set indicates lack of control: the upper and lower warning limits (95% or 2-sigma) and the upper and lower control limits (99% or 3-sigma). Initially, control limits are developed by determining the experimentally estimated standard deviation of approximately 20 independent measurements. Until the experimentally determined control limits are established, the EPA method-specified criteria (if available) are used to assess data quality. The experimentally estimated control limits must be less than or equal to the EPA-specified limits if EPA limits are available. The warning and control limits shall be updated every 20 data points or when a significant change in the measurement system is required to evaluate whether the initial limits are realistic.

The Operations Department staff members prepare and monitor control charts. The QA staff provides oversight in the development of control charts to ensure that they conform to the requirements specified in this policy.

5.4.1 Criteria for Out-of-Control Conditions

The causes for a shift or trend in control charts could result from (1) incorrect preparation of a standard or a reagent, (2) sample contamination (3) improper storage or preservation, (4) incorrect instrument calibration, (5) poor analytical technique, and (6) deviation from the

analytical method. Out-of-control situations may result when one of the following conditions occur:

- A single point outside the control limits.
- A series of seven successive points on the same side of the central line.
- A series of five successive points trending in the same direction
- Any three consecutive points outside of the warning limits.
- A cyclical pattern of control values.

These conditions may indicate that the measurement system is out of statistical control. When this situation occurs, the data will be evaluated thoroughly to identify the most appropriate corrective action to be implemented. The problem and its solution may be documented through an Nonconformance & Corrective Action Record (NCAR) (see Chapter 15) as appropriate. Specifically, in these instances, the data in question must be further examined to identify and correct the root cause of the problem. Exceeding warning limits will only require a close observation of the measurement system. In reviewing control charts, any significant changes in key analysts, instrumentation, or processes must be kept in mind to explain potential out-of-control situations.

5.4.2 Control Analytes

An LCS is prepared by spiking matrix spiking constituents in reagent water matrix (Type II) to represent aqueous samples and in standard solid media (e.g., sodium sulfate for organic analyses) to represent solid matrix samples. For inorganic analyses, a certified PE reference material purchased from an external vendor is used for analysis of solid matrix samples. Results are compared against certified true values (central line) and control limits. An LCS is analyzed for each batch of 20 samples of the similar matrix.

The following constituents will be used to monitor analytical performance via LCS control charts for the methods specified below:

Volatile and Semivolatile Organic Analyses

Three surrogates for volatile organics and six surrogates for semivolatile organics.

Pesticides/PCBs

Two surrogates for pesticides and one Aroclor.

Metals by ICP-AES

At least three control analytes.

Metals by GFAAS

Each analyte (As, Se, Tl, Pb by GFAAS; Hg by CVAAS).

Wet Chemical Methods

Cyanide, Chloride, Nitrate, Sulfate.

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CHAPTER 6
ANALYTICAL INTERNAL QC FOR ANALYSIS OF INORGANIC CONSTITUENTS

6.1 INTRODUCTION

Internal QC is integral to ensuring that analytical results are reliable and that data integrity is maintained throughout the measurement system. Specific guidelines for instrumental calibration are given in the instrument manufacturers' instructions. Guidelines related to sample handling, sample preservation, and sample holding times for inorganic constituents are addressed in SW-846 (EPA, 1986b), in EPA 600/4-79-020 (EPA, 1983), and in Appendix B. This section summarizes the QC activities related to inorganic constituent analyses. These requirements may be superseded by a project-specific QA Plan or a method specific SOP.

At LAS, it is prohibited to alter data solely to meet method- or contract-specified QC requirements.

6.2 HOLDING TIME COMPLIANCE

The method- or client-specified holding time requirements must be met for the sample results to be considered valid. If holding time requirements are exceeded for sample analyses, the client must be informed of the out-of-control situation, sample data must be qualified appropriately, and an explanation of the nonconformity must be provided in the case narrative of the analytical data report.

6.3 DAILY INTERNAL QC FOR EACH ANALYTICAL METHOD

The following internal QC procedures are required for atomic absorption spectroscopy (AAS), inductively coupled plasma-atomic emission spectroscopy (ICP-AES), ion chromatography (IC), and other methods applied to the analysis of inorganic constituents. These requirements may be superseded by a project QA plan or a specific SOP.

6.3.1 Initial Calibration and Instrument Tuning

Method calibrations and/or instrument calibrations must be independently verified daily. Typically, the inorganic methods require the instrument to be calibrated using at least one blank and at least three calibration standards following method SOPs and manufacturer's recommendations for specific procedures. At a minimum, the coefficient of determination (r^2) must be at 0.990, or the correlation coefficient (r) for any given calibration curve must be at 0.995. If the r or r^2 is outside the acceptance criteria, the instrument must be recalibrated. For ICP-AES systems, the instrument must be calibrated according to instrument manufacturer's recommended procedures. Similarly, each ICP system must be calibrated using two standards, one of which must be a blank. All standards used for instrument calibration must be prepared from sources that are traceable to EPA or to the National Institute for Standards and Technology (NIST).

An initial calibration is performed as required for each analytical method (e.g., AAS, ICP-AES). The concentrations of the calibration standards must bracket the expected sample concentrations. The calibration standards must be prepared by using the same type of acid or combination of acids that will be present in the

samples after preparation. All calibration standards for each analytical procedure must be prepared in compliance with the method-specific SOPs. If, during the analysis, the concentration of a sample is above the calibration range or LDR, the sample is to be diluted and reanalyzed. In this case, the diluent must have a matrix similar to the sample matrix with respect to all preservatives (acid type and concentration) used. Samples are first analyzed at the lower concentration range. If a sample concentration exceeds the upper end of the calibration range or LDR, the sample is reanalyzed to fall within the concentration range. Results are reported based on the diluted sample analyses, and the RDLs are adjusted accordingly to reflect the dilutions performed. **Note:** For mercury analysis, the calibration standards must be carried through the acid digestion process.

For inductively coupled plasma/mass spectroscopy (ICP/MS) analysis, each day, the instrument must be tuned by verifying that the system meets the mass calibration and resolution check requirements in the mass regions of interest. If the mass calibration exceeds a difference of more than 0.1 amu from the actual value, then the mass calibration must be adjusted to the correct values. The resolution must be less than 1.0 amu full width at 10 percent peak height. The tuning solution, analyzed at the beginning and end of each analytical run, must have a percent RSD of less than 10 percent. These are required criteria which must be met prior to any sample being analyzed.

6.3.2 Initial Calibration Verification

Immediately after each measurement system (e.g., ICP-AES, AAS, IC) has been calibrated and standardized, the accuracy of the calibration standards and the initial calibration must be verified and documented for each constituent by the analysis of LAS-prepared or certified independent standard solution(s). An independent standard is defined as a standard composed of the same constituents as, but from a different source than, those used in the standards for the initial calibration. If measurements exceed the control limits given in Table 4, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

For ICP-AES, the initial calibration verification (ICV) standard(s) must be run at each wavelength used for analysis. For cyanide, the ICV standard, along with the other samples, must be distilled before analysis. For mercury, the ICV standards must be carried through the acid digestion process.

Furthermore, for ICP-AES analysis using methods 6010 or 200.7, the highest calibration standard must be run as a sample before the analysis to verify the standard concentration. The measured concentration should be within ±5% of the true concentration.

6.3.3 Continuing Calibration Verification

To ensure calibration accuracy during each analytical run, a standard in the mid-range of the calibration curve is analyzed as verification of continued calibration. For most inorganic methods, the continuing calibration verification (CCV) standard must be analyzed at the beginning of the run, at a frequency of 10%, and after the analysis of the last sample. For ICP-AES, the standard must be analyzed for every wavelength used to analyze each constituent. The constituent concentrations in the CCV standard must be a solution at or near the mid-range concentration of the calibration curve and an LAS-prepared standard solution that is independent of the ICV standards.

Table 4. Acceptance Limits for Initial and Continuing Calibration Verification Analyses of Inorganic Constituents

		Percent of True Value	
Analytical Technique	**Constituent**	**Low Limit**	**High Limit**
ICP-AES	6010 and CLP Metals	90	110
	200.7 Metals	95	105
ICP/MS	6020 and 200.8 Metals	90	110
AAS	Metals	90	110
IC	Anions	90 (ICV)	110 (ICV)
		85 (CCV)	115 (CCV)
Cold Vapor AAS	Mercury	80	120
Other	Cyanide	85	115
	Ammonia-Nitrogen	90	110
	Dissolved Silica	90	110
	Fluoride	90	110
	Alkalinity	90	110
	Chromium (hexavalent)	90	110
	pH	-0.1	+0.1

Note: The same continuing calibration standard must be used throughout the analytical runs for each group of samples analyzed.

If the deviation of the CCV exceeds the control limits specified in **Table 4**, the problem must be identified and corrected, the instrument recalibrated, and the preceding 10 analytical samples since the last acceptable calibration verification reanalyzed for the constituents affected.

6.3.4 Laboratory Control Sample

To further ensure that the sample preparation and measurement processes are functioning within control, an independent liquid LCS is prepared or solid LCS purchased from an approved external source. The LCS contains the constituents of interest and is carried through the sample preparation procedure (e.g., digestion, distillation), then analyzed by using the required method. An LCS must be analyzed for each batch of 20 samples of the same matrix. The measured concentrations must fall within 20% of the true concentration or the acceptance limits specified by the vendor. If the percent recovery is outside of the acceptance criteria, all affected samples and the LCS must be redigested and reanalyzed. For solid LCS, data are compared against the advisory control windows specified by the vendor for each analyte. Data obtained for liquid and solid LCSs are further monitored using control

charts. Section 5.4 provides a detailed description of control charts.

6.3.5 Detection Limit QC Standards for AAS and ICP-AES

For ICP-AES, to verify linearity near the IDL, a detection limit QC solution prepared at approximately 2 x RDL is analyzed at the beginning and end of each sample analysis run, or twice per 8-hour work shift, whichever is more frequent. The standard is analyzed for every wavelength used for analysis, except those for ICP-AES analysis of aluminum, calcium, magnesium, sodium, and potassium. For AA, the low-level standard is run only once at the beginning of the analytical run. The measured value is recommended to be within 20 percent of the theoretical concentration; however, specific acceptance criteria has not been established by the EPA. Therefore, data are used for internal data review purposes only.

6.3.6 Initial and Continuing Calibration Blank Analyses

Immediately after every initial and continuing calibration verification, a calibration blank is analyzed (at each wavelength for ICP-AES) and at a frequency of 10% (for most inorganic analytical methods) during an analytical run to check for baseline drift and low-level calibration curve bias.

The calibration blank (theoretically a 0-μg/L standard) contains only the matrix of the calibration standards. The concentration of the analyte in the calibration blank must be less than or equal to the RDL given in Appendix A. If the absolute value of the analyte in the blank exceeds the RDL, the analysis must be terminated, the problem corrected, the calibration checked, and the preceding samples since the last acceptable calibration blank reanalyzed.

6.3.7 Method (Reagent) Blank Analysis

A method blank is a sample that has undergone the same preparation (e.g., extraction, digestion, distillation) procedures as a real sample for analysis. One method blank is processed and analyzed for each group of 20 samples or less of similar matrix for each method that requires sample preparation. Therefore, the method blank results are an indicator of possible contamination. The concentration of the analyte in the method blank must be less than or equal to the RDL (see Appendix A). If the analyte concentration exceeds this limit, the source of contamination must be investigated and if possible, eliminated. All affected samples (having constituent concentration less than 10 times the RDL) in which the high blank value exceeded the RDL of the constituent in question should be reprepared and reanalyzed. If the problem persists, the affected sample datum is qualified appropriately.

It is the policy of LAS not to correct the analytical data for elevated analyte levels in the blank unless otherwise specified by the client or the method.

6.3.8 Matrix (Predigestion) Spike Sample Analysis

The spike sample analysis is designed to provide information about method accuracy and the effect of the sample matrix on the digestion and on measurement methodology. For analytical methods that require sample preparation, the spike is added before sample digestion and/or distillation steps. At least one spiked sample analysis is performed on each group of 20 samples of similar matrix (i.e., aqueous, soil, sludge, or sediment).

The accuracy in terms of percent spike recovery is calculated by using the results of the sample

designated as the "original" sample. Samples identified as blanks or laboratory spikes should not be used for spiked sample analysis because these samples provide minimal information regarding matrix interference.

The required spike level for each constituent analyzed is provided in the analytical method SOP.

If the spike recovery for the matrix spike is not within the limits specified in Appendix A, the LCS data are evaluated to determine if this condition is due to a matrix interference. If the LCS data are acceptable, the data for all samples associated with that spiked sample are qualified as matrix interference in the case narrative. If the LCS results are not acceptable, all samples associated with the batch are reprepared and reanalyzed.

The percent spike recovery (%R) is calculated as follows:

$$\%R = \frac{\text{spiked sample results} - \text{sample result}}{\text{spike added}} \times 100$$

When sample concentration is less than the RDL, a sample concentration of zero can be used to calculate %R.

6.3.9 Duplicate Sample Analysis

One duplicate sample is analyzed from each group of 20 samples of similar matrix.

The within-run precision is calculated as relative percent difference (RPD) between original sample and duplicate sample values as described in Section 3.1.

The acceptance limits for the RPD specified in Appendix A should be used for original and duplicate sample values approximately greater than or equal to 5 times the RDL. For concentrations less than 5x the RDL, the absolute difference between the sample and the duplicate must be less than the RDL.

For Ph determination, precision is expressed as absolute difference between the sample and its duplicate.

If the duplicate sample results are outside of the acceptance limits for a specific matrix, an investigation is done to determine the root-cause of the observed imprecision. Generally, the data are qualified indicating that the sample heterogeneity is suspected.

6.3.10 Graphite Furnace Atomic Absorption QC Analysis

Special procedures are required for quantitation when using the graphite furnace atomic absorption (GFAA) measurement technique. These requirements apply to drinking water analyses and spike analyses.

Drinking Water Analyses - For drinking water analyses, each sample must be spiked with a known concentration of each constituent of interest. The sample is quantitated based on the recovery of the analytical spike.

Analytical (Post-digestion) Spike Analysis - Analytical spikes are added automatically by the instrument during analysis. Analytical spike concentrations are based on specific method requirements.

6.3.11 ICP-AES Interference Check Sample Analysis

To verify interelement and background correction factors, an ICP-AES interference check sample is analyzed at the beginning and

end of each analysis run or at a minimum of twice per 8-hour work shift, whichever is more frequent.

An interference check sample comprises two solutions. Solution A consists of the interferants, and solution AB consists of analytes mixed with the interferants. An interference check sample analysis consists of analyzing the two solutions consecutively (starting with solution A) for all wavelengths used and for each constituent reported by ICP-AES. Table 5 provides the constituent and interferant concentrations used for the ICP-AES interference check samples.

Results of the ICP analyses of solution AB during the analytical runs must fall within the control limit of ±20% of the true value for the constituents included in the interference check samples.

If not, the analysis must be terminated, the problem corrected, the instrument recalibrated, and all analytical samples analyzed since the last acceptable check sample reading reanalyzed.

6.3.12 Internal Standards (ICP/MS)

For ICP/MS, internal standards are used to monitor and correct for changes that occur between standards and samples as a result of physical interferences. A minimum of three internal standards must be selected to bracket the mass ranges 1-70, 71-125, and 126-250. The internal standard must be added to every samples and intensities of any internal standard must fall between 60 to 125 percent (EPA Method 200.8) and 30 to 120 percent (EPA Method 6020) of that internal standard in the initial calibration standard. If recoveries are unacceptable, a different suitable internal standard must be selected, samples must be diluted and reanalyzed, or a higher level spike must be prepared and analyzed.

6.3.13 ICP-AES Serial Dilution Analysis

If the client requires CLP-level analyses, for all constituents analyzed by ICP-AES, the results of the ICP-AES serial dilution analysis on each group of samples of a similar matrix type should be analyzed and reported. A serial dilution analysis involves performing a five-fold dilution on a given sample. Blank samples are not used for serial dilution analysis.

If the constituent concentration is sufficiently high (at a minimum, a factor of 50 times above the IDL in the original sample), an analysis of five-fold dilution must agree within 10% of the original determination. If the % difference exceeds the 10% criteria for an analyte, chemical or physical interference is suspected. Results for that analyte for all samples associated with the batch are qualified appropriately.

The percent difference is calculated as follows:

$$\% \text{ Difference} = \frac{\text{abs } (I - S)}{I} \times 100$$

where I is the initial sample result and S is the serial dilution result (5 times the instrument reading).

6.3.14 ICP-AES Linear Range Analysis

Linear range determination must be performed quarterly for each constituent. The standard must be analyzed during a routine analytical run. The concentration of this standard must be within 5% of the true concentration. This concentration represents the upper limit of the ICP linear range. If measured sample concentrations exceed this level, the samples in question must be diluted and reanalyzed within the linear range.

Table 5. Constituent and Interferant Concentrations Used for ICP-AES Interference Check Sample

Constituent	Concentration (mg/L)	Interferant	Concentration (mg/L)
Barium	1	Aluminum	500
Beryllium	1	Calcium	500
Cadmium	2	Iron	200
Chromium	1	Magnesium	500
Cobalt	1		
Copper	1		
Lead	2		
Manganese	1		
Nickel	2		
Silver	2		
Vanadium	1		
Zinc	2		

6.3.15 ICP-AES Interelement Correction Determination

On an annual basis, correction factors for spectral interference due to aluminum, calcium, iron, and magnesium must be determined for all ICP instruments at all wavelengths used for each constituent.

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CHAPTER 7
ANALYTICAL INTERNAL QC FOR ANALYSIS OF ORGANIC CONSTITUENTS

7.1 INTRODUCTION

Quality control is integral to ensuring that analytical results for organic constituents are reliable and that data integrity is maintained throughout the measurement system. Specific guidelines for instrumental calibration and tuning and for methods of sample handling, sample preservation, and holding times are described in LAS method-specific SOPs, instrument manufacturers' guidelines, and EPA methods. This section summarizes the QC activities related to organic constituent analyses. These requirements may be superseded by a project-specific QA Plan or a method-specific SOP.

At LAS, it is prohibited to alter data solely to meet method- or contract-specified QC requirements.

7.2 HOLDING TIME COMPLIANCE

The method- or client-specified holding time requirements must be met for the sample results to be considered valid. If holding time requirements are exceeded for sample analyses, the client must be informed of the out-of-control situation, sample data must be qualified appropriately, and an explanation of the nonconformity must be provided in the case narrative of the analytical data report.

7.3 SYSTEM TUNING OF THE GC-MS SYSTEM

It is necessary to establish that a given gas chromatograph-mass spectroscopy (GC-MS) system meets the standard mass spectral abundance criteria specified in the method before sample analysis begins. This system tuning is accomplished through the analysis of p-bromofluorobenzene (BFB) for volatile organic analyses and decafluorotriphenylphosphine (DFTPP) for semivolatile organic analyses.

Before any sample, blank, or standard is analyzed, the hardware for each GC-MS system must be tuned to meet the method-specified ion abundance criteria. The ability to meet the abundance criteria must be demonstrated for each 12-hour period, unless otherwise specified. Whenever corrective action is taken that may affect the tuning condition (e.g., ion source cleaning or repair), the tune must be verified regardless of the 12-hour tuning requirements. If background subtraction is required, it must be designed to eliminate interferences that may result from column bleed or instrument background ions, and must not be performed solely to meet QC requirements. The documentation of the tuning is provided as a bar graph spectrum and as a mass listing.

7.4 INITIAL CALIBRATION OF THE ANALYTICAL SYSTEM

7.4.1 Initial Calibration of the GC-MS System

Before samples and required blanks are analyzed and after tuning criteria have been met, calibration standards that contain all the target compounds, surrogates, and internal standards are analyzed at method-required concentrations to calibrate the GC-MS initially to determine the sensitivity of the system and the linearity of response. Once the system has been calibrated, the initial calibration must be verified every 12

hours, unless otherwise specified, for each GC-MS system.

Secondary ion quantitation should be performed only when there are sample interferences with the primary ion. All standards shall be analyzed under the same conditions as the method blank and the routine samples. The relative retention times of each constituent in each calibration run should agree within method-specified criteria.

The initial calibration is considered valid only after the minimum relative response factor (RRF) and the %RSD criteria have been met. Sample analysis can begin only after these criteria have been met. When the accuracy of initial calibration is verified, the average RRFs and the %RSD for all target compounds must be calculated and reported on the appropriate calibration summary sheet.

7.4.2 Initial Calibration of the GC, HPLC, and FT-IR Systems

For the gas chromatography (GC), high performance liquid chromatography (HPLC), and Fourier transform infrared spectrophotometry (FT-IR) systems, the following calibration procedures must be performed. Before samples and required blanks are analyzed, calibration standards that contain all the target compounds and required surrogates are analyzed at method-required concentrations to calibrate the analytical system initially to determine the linearity of response.

If the method specifies the use of response factors for compound quantitation (i.e., CLP), the %RSD for each compound must meet the method-specified criteria. If %RSD is exceeded, an appropriate corrective action is instituted and a new initial calibration is performed.

Other methods, particularly SW-846, allow the analyst to use a calibration method best suited to the analytical technique. The calibration method used primarily at LAS is a quadratic fit forced through zero, followed by linear regression forced through zero, and linear non-forced through zero. The coefficient of determination (r^2) must be greater than or equal to 0.990 or the correlation coefficient (r) must be greater than or equal to 0.995 for the calibration to be considered valid in determining constituent concentration. Point-to-point calibrations are not used at LAS.

7.5 RETENTION TIME WINDOWS

Identification of target analytes is achieved by the use of retention time windows. For methods (i.e., GC/MS methods) that use internal standards, this is accomplished by establishing the retention time of each analyte relative to the internal standard during the daily continuing calibration. Relative retention times (RRTs) of target analytes are then calculated for all subsequent analysis. The RRT criteria must be met for analyte identification to be considered accurate.

External standard methods (i.e., GC, HPLC, FT-IR methods) differ in that the retention time criteria are established by using the absolute retention time for each analyte established during the initial calibration. A retention time window is established from the absolute values. The retention time window criteria may vary depending on the method. LAS' method-specific SOPs detail the requirements for establishing retention time windows for applicable methods.

7.6 INTERNAL STANDARDS

For the methods that employ internal standards for target constituent quantitation (i.e., GC/MS

methods), the internal standard solution must be added to every standard, blank, matrix spike, matrix spike duplicate, sample (for VOAs) and sample extract (for semivolatiles analyses).

Internal standard responses and retention times in all standards must meet the method-specified criteria. If these criteria are not met, the chromatographic and/or the mass spectrometric system must be inspected for malfunctions. When corrections are made and the system is demonstrated as in control, the affected samples shall be reanalyzed.

The extracted ion current profile (EICP) and retention times of the internal standards must be monitored and evaluated for each sample, blank, matrix spike, matrix spike duplicate, and LCS. If method-specified criteria were not met, the sample in question must be reanalyzed. If reanalysis does not solve the problem, both analyses results are reported.

7.7 CONTINUING CALIBRATION VERIFICATION

7.7.1 Continuing Calibration Verification of the GC-MS System

A mid-level calibration standard containing all target compounds, required surrogates, and internal standards must be analyzed every 12 hours during analysis, unless otherwise specified, to verify the initial calibration.

The internal standard responses, retention times, minimum RRFs, and percent difference (%D) criteria specified in the method or LAS SOPs must be met for the continuing calibration to be considered valid. If these criteria are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some potential problems may result from standard mixture degradation, purge-and trap system contamination (VOAs only), injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatography system. If the source of the problem cannot be identified after corrective action has been taken, a new initial calibration is required.

7.7.2 Continuing Calibration Verification of the GC, HPLC, and FT-IR Systems

A mid-level calibration standard containing all target constituents and required surrogates must be analyzed at the frequency specified by the method or the client.

Percent difference (%D) of concentration (i.e., the difference between the concentration of the continuing calibration and the midlevel initial calibration standard) must meet method- or client-specified QC criteria for the continuing calibration to be considered valid.

If the %D criterion is exceeded for any analyte, corrective action must be taken. The experience and the professional judgement of the analyst play a key role in determining the most suitable action. If the source of the problem cannot be identified after corrective action, a new initial calibration curve must be generated for the analyte that exceeded the criterion. These criteria must be met before sample analysis is continued.

7.8 LABORATORY CONTROL SAMPLE

An LCS is a volume of reagent water (meeting the specifications for ASTM Type II water or better) for aqueous samples or a contaminant-free solid matrix for soil or sediment samples, which is spiked with known quantities of target analytes and required surrogates. An LCS is prepared independently from the calibration standards and carried through the entire analytical process. An LCS must be analyzed

for each batch of up to 20 samples of the same matrix.

In the event that LCS data exceed the QC limits, the LCS recovery data are evaluated in conjunction with other QC analyses (i.e., MS/MSD, QCCS, surrogate spikes, method blank) to determine if the analytical process is in control. If the process is judged out-of-control, all affected samples and method blanks must be reextracted and reanalyzed.

7.9 INSTRUMENT AND METHOD BLANK ANALYSIS

An instrument blank consists of deionized, distilled water spiked with surrogates and is carried only through the analytical process. For low-level volatile organic analyses, instrument blank serves as a method blank because no preparation procedure is required for this method. The instrument blank measures any contamination that may result during analysis. The instrument blanks must be analyzed at a frequency specified by the method.

A method blank consists of all reagents and required surrogates in a volume of deionized, distilled laboratory water (meeting the specifications for ASTM Type II water or better) for aqueous samples or in a contaminant-free solid matrix for soil or sediment samples. However, the method blank is carried through the entire analytical process (i.e., extraction, concentration, and analysis). Its volume or weight must be approximately equal to the sample volumes or sample weights being processed.

A method blank must be analyzed for each batch consisting of up to 20 samples. For the analysis of volatile target constituents, a method blank must be analyzed before sample analysis, within each 12-hour period, or for each 10 samples, as specified in the method. Laboratory personnel must ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample-processing hardware that lead to discrete artifacts or elevated baselines are minimized.

No contaminants shall be detected above the RDLs in the instrument and method blanks. If a blank exceeds the RDL, the source of the contamination is investigated, and appropriate corrective actions are taken and documented before sample analysis proceeds. All samples associated with the method blank that contain high target constituent(s) must be reextracted/reanalyzed or the affected sample results properly qualified. The measured concentration of common laboratory contaminants (i.e., acetone, methylene chloride, and phthalates) must not exceed five times the RDL. Otherwise the samples associated with the unacceptable blanks must be reextracted and reanalyzed (Semivolatile organics) or reanalyzed (VOAs).

It is the policy of LAS not to correct the analytical data for elevated analyte levels in the blank unless specified by the client or method.

7.10 SURROGATE SPIKE ANALYSIS

The surrogate standards, specified in each method-specific SOP, are to be added to each sample, blank, duplicate, LCS, matrix spike, and matrix spike duplicate before purging or extraction. The surrogate spike recovery data provide information regarding the efficiency of the sample preparation and the analytical process. Surrogate analysis is evaluated by determining whether the surrogate spike percent recovery (measured as concentration) falls within the acceptance criteria for each method.

7.10.1 Surrogate Spike Recovery in Method Blanks

If recovery of surrogate compound in the method blank exceeds QC limits, the problem must be investigated to identify the root cause. If the extraction and/or analytical process is judged to be out-of-control and sample data quality is adversely affected, all affected samples shall be reextracted and reanalyzed when sufficient sample aliquots are available and holding times are not expired. The problem must be corrected before sample analysis proceeds. The specific corrective action is determined by the instrument operator and his/her technical lead and supervisor.

7.10.2 Surrogate Spike Recovery in Samples

If recovery of surrogate compound in client sample exceeds QC limits, the problem must be investigated to identify the root cause. The surrogate results in question must be evaluated in conjunction with other QC data (i.e., LCS data and method blank data). The same sample extract may be reanalyzed to determined if the out-of-control condition resulted from isolated, poor instrument performance. If the extraction and/or analytical process is judged to be out-of-control and sample data quality is adversely affected, the sample in question shall be reextracted and reanalyzed when sufficient sample aliquots are available and holding times are not expired. The problem must be corrected before sample analysis proceeds. The specific corrective action is determined by the instrument operator and his/her technical lead, supervisor, and the project manager.

7.11 MATRIX SPIKE AND MATRIX SPIKE DUPLICATE ANALYSIS

The matrix spike analysis is designed to provide information about the effect of the sample matrix on the measurement methodology. The minimum QC requirements for matrix spike and matrix spike duplicate analyses are listed as follows:

- A matrix spike analysis and a matrix spike duplicate analysis must be performed for each batch of up to 20 samples of a similar matrix and processed through the same procedure. Prepare the matrix spike solutions according to the method-specific SOPs.

- The percent spike recovery for matrix spike/matrix spike duplicate and relative percent difference (RPD) between the matrix spike and matrix spike duplicate are calculated as specified in the method-specific SOPs.

- If the QC criteria are not met for matrix spike or matrix spike duplicate results, use the results in conjunction with other QC data (i.e., LCS results, surrogate data, internal standard response) and determine the need for corrective action.

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CHAPTER 8
ANALYTICAL INTERNAL QC FOR ANALYSIS OF RADIOCHEMICAL CONSTITUENTS

8.1 INTRODUCTION

Internal QC is integral to verifying that data integrity is maintained throughout the measurement process. The validity of the radionuclide data generated can only be ensured through accurate and precise instrument calibration and through implementing rigorous internal QC practices. This section summarizes the QC activities related to radiochemical constituent analyses. The types and uses for various QC samples are also described in tables 2 and 3 in Chapter 3. The requirements in this chapter may be superseded by a project QA plan or a specific SOP.

At LAS, it is prohibited to alter data solely to meet method- or contract-specified QC requirements.

8.2 HOLDING TIME COMPLIANCE

The method- or client-specified holding time requirements must be met for the sample results to be considered valid. If holding time requirements are exceeded for sample analyses, the client must be informed of the out-of-control situation, sample data must be qualified appropriately, and an explanation of the nonconformity must be provided in the narrative of the analytical data report.

8.3 CALIBRATION OF THE COUNTING INSTRUMENT

The calibration of detection instrumentation used in radiochemical determinations, including initial setup and method-specific calibrations, is time consuming. Unlike calibration of other analytical measurement systems, however, calibration of radiochemical instruments is stable for long durations. Nevertheless, each instrument shall be calibrated and checked for drift as specified in the instrument manual, EPA and other nationally recognized standards, and practical laboratory experience. If more restrictive calibration requirements are requested by the client, calibrations will be performed according to project specific requirements when counting the project samples. All QC requirements regarding calibration must be met before sample analysis can proceed.

The calibration standards must be prepared or obtained from NIST-certified standards, NIST-traceable commercial standards, standards available from EPA, or commercial standards traceable to a national laboratory equivalent to NIST. The activity of each source shall be certified by the manufacturer, including uncertainty of the measurements.

8.3.1 Gross Alpha and Beta Counting by Gas Proportional Detector

8.3.1.1 Initial Instrument Setup and Calibration. The instrument must include a low-background, anticoincidence proportional counter, a sample detector, and cosmic detector. It must also discriminate between alpha and beta pulses. The instrument is to be configured according to the manufacturer's instructions; any changes or modifications to the configurations must be documented in the appropriate SOP.

Before samples and required blanks are counted, the instrument must be initially calibrated to establish operating high voltage, adjust for proper alpha and beta separation, and determine

alpha and beta background count rate, and alpha and beta counting efficiencies. Standard sources Am-241, Tc-99 and other sources as required by the project shall be used for calibration.

The plateau voltage is determined by counting one standard over a range of voltage in increments. Plot voltage versus counting activity to determine proper operating voltages for both alpha and beta counting. After plateau voltage is set, cross talk (i.e., sensitivity) and counting efficiency shall be determined. Cross talk shall be determined by counting known alpha activity on a beta counter, and known beta activity on alpha counter. Counting efficiency shall be determined by counting a known activity for each counting geometry over the sample weight range expected to be encountered during analysis.

LAL-91-SOP-0078 and LAL-91-SOP-0079 provide instructions and guidance for the calibration, maintenance, and operation of the Tennelec Alpha/Beta counting systems employed at the LAS. All QC criteria specified in the SOPs regarding initial instrument setup and calibration must be met before sample analysis can proceed. The instrument shall be recalibrated per the appropriate SOPs and instrument manuals after repair, change of detector, or when continuing calibration verification cannot meet QC requirements.

8.3.1.2 Calibration Verification. Background counts of at least 1-hour duration must be performed daily on each detector. Duration of the background count should be the same as the expected sample count duration. If the background counts are greater than two times the long-term average, the instrument is considered out of control.

Standard alpha and beta sources must be counted on each detector on daily basis. A minimum of 10,000 net counts shall be acquired for Tc-99, and a minimum of 10,000 net counts shall be acquired for Am-241. The value must fall within the 3 standard deviations of a long term mean value.

A plateau count shall also be performed whenever there is concern regarding the integrity of the system, as indicated by background and check source counts, to verify whether the initial calibration is valid.

A new initial calibration shall be performed when calibration verification checks cannot meet SOP or project specified QC requirements.

8.3.2 Alpha Spectroscopy System

8.3.2.1 Initial Instrument Setup and Calibration. The alpha spectroscopy system shall consist of a detector system capable of measuring alpha isotopes in the range of 3 to 7 MeV. The system shall have a resolution of less than 50 KeV for the isotopes Am-241 (5.49 MeV) or Pu-239 (5.14 MeV).

Before samples and required blanks are counted, the instrument must be initially calibrated for energy and counting efficiency by using multi-point alpha standards. A multinuclide source containing two or more nuclides such as Am-241, Cm-244, and Pu-239, or equivalent, shall be used for calibration.

LAL-91-SOP-0077 provides instruction and guidance for the calibration, maintenance, and operation of the alpha spectrometry system. All of the QC criteria specified in the SOP regarding initial instrument setup and calibration must be met before sample analysis can proceed. The instrument shall be recalibrated per the appropriate procedures and instrument manuals after repair, maintenance to the detector or electronics, and when continuing calibration verification cannot meet QC requirements.

8.3.2.2 Calibration Verification. Background counts of at least 12-hour duration for all regions of interest in use must be performed on a weekly basis for each detector. Duration of the background count should be the same as the expected sample count duration. If the background counts are greater than the project specific MDA's, the instrument is considered out of control.

Instrument check standards shall be counted once a week or prior to sample analysis, whichever is less frequent, to verify energy and efficiency calibration for a minimum of two nuclides. A minimum of 2,000 counts in each peak shall be acquired to compromise between the probability of detector contamination and counting statistics. The value must fall within the 3 standard deviations of a long term mean value.

If the calibration verification check exceeds the control limits, no samples are counted until the problem is investigated and the instrument is brought back into control. A new initial calibration shall be performed when calibration verification checks cannot meet SOP or project specified QC requirements.

8.3.3 Liquid Scintillation Counting System

8.3.3.1 Initial Instrument Setup and Calibration. A low-background counter consisting of two photomultiplier tubes that recognize coincidence events is required. The counter is operated with windows that maximize the figure of merit (FOM) when the samples are counted. The calibration standards are used to set the windows.

Before samples and required blanks are counted, the instrument must be initially calibrated for counter windows, counting efficiency and quench curve. The counter shall be calibrated with windows that maximize the FOM when possible (i.e., FOM = E^2/B; where E = detector efficiency, and B = background count per minute [CPM]).

A quench curve shall be established for each radionuclide of interest over a range of varying quenching similar to that is normally encountered during analysis. At least 10,000 counts are accumulated for each unquenched and quenched standard. For quench curve determination, both internal and external quench methods are used for at LAS. The external method is used routinely because it is more efficient for large batches of samples. The internal method (i.e., matrix spikes) is used as a verification of the external quench determination and when small numbers of samples make generating the external quench curve less efficient. The quench standards for each radionuclide to be analyzed shall be prepared according to the procedure specified in the corresponding method SOP for that radionuclide. The quench standards shall be contained in the same type of scintillation vial to be used for sample analysis.

LAL-91-SOP-0080 and LAL-91-SOP-0081 provide instructions and guidance for the calibration, maintenance, and operation of the liquid scintillation counters employed at the LAS. All of the QC criteria specified in the SOPs regarding initial instrument setup and calibration must be met before sample analysis can proceed. The instrument shall be recalibrated per the appropriate procedures and instrument manuals after repair, maintenance to the detector or electronics, and when continuing calibration verification cannot meet QC requirements.

8.3.3.2 Calibration Verification. Background counts of at least 1-hour duration must be performed on a daily basis. If the background counts exceeds the 3 standard deviations of a long term mean value, the instrument is considered out of control.

Standard sources of H-3 and C-14 shall be counted daily. A minimum of 100,000 net counts shall be acquired. The value must fall within the 3 standard deviations of a long term mean value.

If the calibration verification check exceeds the control limits, no samples are counted until the problem is investigated and the instrument is brought back into control. A new initial calibration shall be performed when calibration verification checks cannot meet SOP or project specified QC requirements.

8.3.4 Gamma Spectroscopy System

8.3.4.1 Initial Instrument Setup and Calibration. A beta absorber consisting of about 6 mm of aluminum, beryllium, or plastic may be used for samples that have a significant beta activity and high beta energies. Germanium detectors with high resolution are used for gamma spectrometry. The detector output is digitized and stored by using a multichannel analyzer. The system must be set up according to manufacturer's specifications.

Before samples and required blanks are counted, the instrument must be initially calibrated for energy and counting efficiency.

Depending upon the intended use, the gamma spectroscopy system should be calibrated for different energy ranges. In general, the energy range is set from 50 KeV to 2000 KeV which is suitable for most applications. However, other energy ranges may be utilized as necessary to analyze nuclides with energies lower or higher then this range.

For energy calibration, use an NIST traceable standard source containing a gamma emission near the lower and upper end of the desired energy range. For the 50-2000 KeV range, the mixed source SRM4275, containing the 123.14 KeV and 1274.5 KeV europium-154 peaks, may be used. Each photopeak of interest in the final spectra must contain no less than 10,000 net counts. A two-point calibration on the spectra is required to generate calibration data for energy versus channel number and the peak shape (FWHM) versus energy number.

Efficiency calibrations should be performed over the range of matrices and geometries normally encountered to obtain attenuation curves for each procedure. After the energy calibration is completed for the system, collect an energy spectrum using a calibrated radioactivity standard in a desired and reproducible counting geometry. The efficiency calibration may be performed using one or more spectra to obtain the required numbers of isolated singlets over the entire energy range of interest. Each full-range gamma-ray peak of interest must contain at least 10,000 net counts.

LAL-91-SOP-0075 provides instruction and guidance for the calibration, maintenance, and operation of the Gamma Spectroscopy System. All of the QC criteria specified in LAL-91-SOP-0075 regarding initial instrument setup and calibration must be met before sample analysis can proceed. The instrument shall be re-calibrated per the appropriate procedures and instrument manuals after repair, maintenance to the detector or electronics, and when continuing calibration verification cannot meet QC requirements.

8.3.4.2 Calibration Verification. Background counts of at least one hour duration shall be performed on each detector on no less than a weekly basis. Background count duration shall be as long as the longest sample count duration. The background counts must fall within the three standard deviation of the long term average of the LLD.

A three-point energy and efficiency calibration verification shall be performed daily using a sealed check source. The source used shall

meet the SOP specifications. The peak centroid energy, FWHM, and counting efficiency must fall within the 3 standard deviation uncertainty limits for each of the three peaks.

If the calibration verification check exceeds the control limits, no samples are counted until the problem is investigated and the instrument is brought back into control. A new initial calibration shall be performed when calibration verification checks cannot meet SOP or project specified QC requirements.

8.4 METHOD BLANK ANALYSIS

A method blank is a sample composed of all the reagents (in the same quantities) in the reagent-grade water carried through the chemical separation process and are used to determine sample contamination introduced during sample preparation.

Method blanks are analyzed at a frequency of 5% per batch and are analyzed in the same manner and with the same aliquot and count time as the samples. As required to obtain a statistically significant number of counts, the reagent blank may be counted longer than the samples.

The method blank value must be less than or equal to two times the MDA (or the RDL). If the reagent blank data indicate an out-of-control condition, the cause of the contamination must be eliminated **before** more samples are analyzed.

The samples counted with the contaminated blanks must be qualified or reprepared and reanalyzed, depending on the customer's needs.

It is the policy of LAS <u>not</u> to correct the analytical data for elevated radionuclide levels in the blank unless specified by the client or method.

8.5 LABORATORY CONTROL SAMPLE

To ensure that the sample preparation and counting processes are functioning within control, an independent LCS is prepared for each batch containing up to 20 samples. LCSs must be prepared and analyzed in the same manner as the samples, and must have the same aliquot size and count time as the samples. In the absence of absorption corrections and yield, the LCS will have the same MDA as the sample.

The LCS data must meet the SOP- or project-specified QC limits. The samples results generated with the out-of-control LCS shall be qualified or the sample reanalyzed, depending on the requirements of the customer.

8.6 CHEMICAL YIELD

Method performance on individual samples subject to chemical process and separation is established by means of spiking with tracer which is a radionuclide of the same element of the radionuclides of interest or with stable carrier of the same or a chemically similar element. All samples and QC samples shall be spiked prior to sample preparation. Sample specific chemical recoveries must meet the SOP- or project-specified QC requirements for LCS and method blank samples which are free from matrix interference.

Since the effects of sample matrix are frequently outside the control of the laboratory and may present relatively unique problems associated with each sample, the evaluation of data quality will be performed based on other QC results (i.e., LCS, method blank and duplicate recoveries), analytical experience, and professional judgement. If QC limits are not met for sample analysis, an explanation shall be provided in the case narrative of the final report.

8.7 DUPLICATE SAMPLE ANALYSIS

One duplicate sample shall be performed for each 10 samples (i.e., at a frequency of 10%) in a batch. Duplicate sample must be analyzed in the same manner and with the same aliquot and count time as the sample. Samples identified as field blanks shall not be used for duplicate sample analysis because poor precision is expected near the MDA. The method precision is determined as per SOP or project specifications.

If the duplicate analysis exceeds QC limits, the matrix homogeneity shall be evaluated to determine if reanalysis is required. If the duplicate analysis is out-of-control, a second, different sample, of the similar matrix, may need to be analyzed in duplicate or data must be qualified and a narrative shall be attached to the analysis batch worksheet.

8.8 MATRIX SPIKE AND MATRIX SPIKE DUPLICATE ANALYSES

Matrix spike (MS) and matrix spike duplicate (MSD) samples are prepared and analyzed only as required by the client. These samples are used to determine the sample matrix effect on the accuracy and precision of the measurement process.

The percent spike recovery and duplicate precision calculated as per SOP or project-specific requirements must meet the QC limits specified by the SOP or project. If the QC criteria are not met for MS or MSD results, the results from other QC analyses (i.e., LCS, duplicates, and other chemical recovery) are evaluated to determine the extent of corrective action.

CHAPTER 9
SYSTEMS AND PERFORMANCE AUDITS

9.1 INTRODUCTION

Technical systems audits, management assessments, and performance evaluations are essential in every quality assurance program. These audits are used to determine on-going compliance with the quality assurance program and project plans and to assess the overall quality of data collected during the measurement process. Furthermore, audits help in evaluating sample collection, sample analysis, and data handling procedures. The objectives of these audits are (1) to confirm proper conduct of all sample handling, sample analysis, and data handling and reporting procedures and (2) to minimize the generation of invalid data by detecting potential problems at the earliest stage possible in these processes. Such practices can save time and reduce costs associated with resampling and reanalysis.

9.2 TECHNICAL SYSTEMS AUDITS

9.2.1 Internal Audits

A technical system audit is an in-depth, qualitative, on-site evaluation of a sample handling, measurement, and data handling system. These audits are accomplished through (1) observing project activities, (2) inspecting operating conditions and documentation, and (3) interviewing project participants. The primary objective of an internal system audit is to assess and document all facets of the measurement process: facilities, sample preparation, instrument operation, instrument calibration, analytical measurement, data generation, data validation, data reporting, document control, waste handling, and overall QC practices.

These evaluations enable LAS management to ensure that three important actions are being performed:

- Record keeping is implemented in accordance with LAS Notebook Policy (LAL-90-SOP-0006) and sample chain-of-custody activities are implemented in accordance with Sample Receiving and Login (LAL-90-SOP-0002) and LAS Internal Chain-of-Custody and Evidentiary Procedures (LAL-90-SOP-0009).

- Current versions of SOPs (Appendix C) are readily available, properly controlled, and are being followed by the laboratory staff.

- Analytical QC is being followed in accordance with the LAS Quality Assurance Management Plan, the method or SOP, and all other client specifications. For this purpose, analytical QC is defined as (1) the analysis of the proper types and numbers of QA and QC samples (e.g., standard reference or performance evaluation samples, blanks, spikes, duplicates), (2) the maintenance of proper standards traceability, (3) the data reporting from those analyses on laboratory reporting forms, and (4) the use of the proper corrective action measures.

In order to eliminate any question of conflict of interest, the technical system audits of analytical processes must be performed by quality assurance personnel or other independent Lockheed staff who have no other responsibilities related to the data generation operations and who report outside the Operations Department. At the

LAS, auditors are part of the Quality Assurance Department; they report directly to the Quality Assurance Manager who, in turn, reports directly to the LAS Director (see Chapter 2). LAL-90-SOP-0010 provides details of the LAS approach to internal technical systems auditing.

9.2.1.1 Systems Audit Procedures. During the audit, the auditor may use the LAS laboratory technical systems evaluation questionnaire to document observations. This process ensures that the auditor has examined all elements of the system under evaluation. The questionnaire also aids in discussing data quality issues related to the QA and QC sample analyses (e.g., blank, spike, duplicate, and single-blind samples). Documentation, such as chain-of-custody forms, analysis request forms, SOPs, logbooks, reagent bottle labeling, and instrument printouts, is inspected and randomly cross checked (e.g., dates, initials) when applicable. A detailed discussion of the technical systems evaluation is given in LAL-90-SOP-0010.

The auditor summarizes all observations in a technical systems evaluation report and brings all problems observed to the attention of the Quality Assurance Manager, Operations Manager, and responsible supervisors for corrective action. The auditor also maintains a log used to track corrective action requests and results. Chapter 15 provides details of the corrective action mechanisms in place at LAS.

9.2.1.2 Internal Auditing Schedule. Routine technical systems evaluations are performed semiannually. These evaluations may or may not be announced to the operations staff. Furthermore, unannounced follow-up evaluations are performed as required to ensure that any deficiencies identified during the routine evaluations were corrected in a timely manner. In addition, preliminary systems audits may be performed for major clients before any environmental samples are analyzed for a particular project to ensure that the laboratory is generating products of an acceptable quality.

9.2.2 External On-site Systems Audits

It is LAS policy to respond in writing to any corrective action requirements identified by external auditors. The response includes the corrective actions implemented, or the proposed resolution and the proposed schedule for its implementation.

9.3 PERFORMANCE EVALUATIONS

9.3.1 Internal Performance Evaluations

A performance evaluation (PE) audit is a quantitative evaluation of the laboratory analytical system. PE audits are performed annually or more often as new major methods are introduced into routine operations. The evaluation generally involves the measurement of a PE reference material that has a known value or composition. These samples are key factors in environmental sample analysis; thus, they must be of high quality. It is important that the reference material be certified (e.g., NIST, EPA, private supplier) or, at a minimum, verified before use, and that the certification or verification be adequately documented. Certification documents are maintained by the Quality Assurance Department.

The Quality Assurance staff ensures that the PE materials are selected in such a way that the concentration of PE samples are representative of the media and the levels of inorganic, organic, and radionuclide constituents typically processed at LAS.

Two types of PE samples are used to monitor analytical system performance, such as single-blind and QC check standards (QCCSs). Single-blind PE samples are samples that the

analyst knows are audit samples, but for which the analyst does not know the constituent levels. QCCS are samples submitted in such a manner that the analyst knows that the sample is a PE sample and is also aware of the theoretical concentration of the constituents in the sample. The main function of the QCCS is to provide immediate feedback to the analyst during the sample analysis, so that if the results of the QCCS analysis do not fall within predetermined levels of precision or accuracy, appropriate corrective actions within the analysis system can be pursued.

Data obtained from the analyses of the audit samples are used for the following purposes:

- To judge the ongoing capability of the analyst and sample preparation technician, the reliability of the instrumentation, and the proficiency of the method(s).

- To establish a statistically valid estimate of the accuracy and precision of the measurement system.

- To assess whether or not the system is operating within the established control limits on a daily basis and over extended periods.

Acceptance criteria may be established for the measurement of each constituent in the audit samples. The recommended acceptance limits provided by the PE material supplier and method-specified QC acceptance criteria are used to assess the acceptance of data. If the analytical results fall outside the criteria, the Quality Assurance auditor documents this condition on the Nonconformance & Corrective Action Record and immediately contacts the analytical laboratory personnel to request corrective action. Typically, corrective actions may require (1) recheck and recalculation of data, (2) reevaluation of other related QC results, or (3) instrumental or procedural refinements. If major deficiencies are identified, another suitable PE material may be submitted to verify that the proper actions have been executed to eliminate or minimize the potential for recurrence of the problem.

9.3.2 External Intercomparison and Performance Evaluation Studies

Semiannually, LAS participates in the analysis of water pollution (WP) and water supply (WS) PE samples issued by the EPA EMSL-Cincinnati as required by the Clean Water Act (CWA) and Safe Drinking Water Act (SDWA) Programs. The results achieved from these studies verify our capability to analyze low-level, drinking water and waste water samples for inorganic and organic constituents.

As specified by the LAS Radioactive Material License issued by the Nevada State Health Division, LAS consistently and actively participates in U.S. EPA's Environmental Radioactivity Laboratory Intercomparison Studies Program. Simulated environmental performance evaluation (PE) samples containing known amounts of one or more radionuclides of interest are analyzed to verify LAS' capability to determine low-level radionuclides in multimedia environmental samples (e.g., water, air, vegetation, milk, etc.) with a stated level of confidence. The LAS also participates in the Department of Energy's (DOE's) Environmental Measurement Laboratory (EML) Radiochemical Proficiency Program for the analysis of various radionuclides in environmental and low-level mixed waste samples that consist of water, soil, and vegetation. In addition, each year, LAS analyzes PE materials to obtain accreditations through various federal, state, and local accrediting authorities (e.g., U.S. Army Corps Of Engineers, State Departments of Health).

The analyses of these PE samples independently demonstrates LAS' capability to perform required testing and to produce analytical results of known and documented quality.

Table 6 presents the external PE Programs administered by the EPA and DOE in which LAS routinely participates.

9.4 MANAGEMENT ASSESSMENTS

Each year, the LAS Director or designated management staff evaluates the LAS Quality Assurance Program to ensure its continuing suitability and effectiveness of its implementation and to introduce any necessary changes or improvements. The purpose of this independent, qualitative assessment is to verify the effectiveness of the LAS Quality System by determining the adequacy of policies, objectives, organization, procedures, and practices to ultimately ensure data quality. This type of an assessment provides a management tool for continuous evaluation and improvement of LAS activities. This assessment typically includes a detailed review of the following matters:

- Results from external on-site audits conducted by clients.
- Results from internal evaluations, including corrective actions implemented.
- Results of PE intercomparison studies.
- Results of internal blind PE studies.
- Details of any complaints from clients.
- Staff training for new and existing staff.
- Adequacy of staff, equipment, and facility resources.

The management assessment findings and actions are documented in the form of an internal memorandum.

Table 6. External PE Intercomparison Studies in Which LAS Routinely Participates

Agency	PE Study Type
U.S. EPA Environmental Monitoring Systems Laboratory at Cincinnati, OH	Water Pollution/Water Supply for inorganic and organic constituents
U.S. EPA Environmental Monitoring Systems Laboratory at Las Vegas, NV	Uranium in water Alpha, Beta in water Mixed Gamma in water Pu-239 in water Ra in water
DOE Environmental Measurements Laboratory (EML)	Uranium in soil Uranium in water Mixed gamma in water and soil Total U in water and soil Pu in water and soil
U.S. Army Corps Of Engineers	Inorganic and organic constituents

CHAPTER 10
DATA HANDLING, MANAGEMENT, AND REPORTING

10.1 INTRODUCTION

A carefully executed Quality Assurance Program emphasizes sufficient document control and data management, minimizes the generation of data that are not scientifically or legally defensible, and results in efficient, cost-effective data management. Data needs differ depending on the requirements of a specific project or client. Rigorous, broad-based analysis, QA, and QC are especially important in cases involving site clean-up because legal actions rely heavily on the quality of analytical data generated during site characterization. For environmentally related measurements, the quality assurance program established for the CLP is considered to be the minimum necessary to provide defensible data for regulatory, enforcement, legal, or policy matters. At LAS, proper documentation is an important facet of scientifically sound, high-quality data.

The National Enforcement Investigations Center (NEIC) in Denver, Colorado, has established policies and procedures for data handling in analytical laboratories that participate in the CLP (Laidlaw, 1986). The significance of NEIC enforcement is that data and documents are evidentiary materials and, as such, must be able to withstand legal scrutiny. NEIC policies and procedures have been incorporated into the data handling operations at LAS.

10.2 DATA CONFIDENTIALITY

It is LAS policy to preserve the confidentiality of data and reports generated by the laboratory and to respectfully decline release of this information to persons other than authorized representatives of clients. Reports and supporting records maintained in the Document Control Section are not released to persons without approval from laboratory management. Furthermore, records are not released to persons or organizations outside of Lockheed unless directed to by competent authority in the client's organization. If directed by courts-of-law or other competent authorities, such as regulatory agencies, we will provide records as necessary and notify our clients and provide information as to the identification of the requestor and the records that were released.

It is also our policy to respect client and/or project requirements for confidentiality of projects. When confidentiality clauses are contained in contractual documents, we will not release any information without first obtaining written approval from the client.

10.3 SAMPLE RECEIPT DATA

Chapter 4, Sample Management, and Chapter 13, Internal Sample Chain-of-Custody and Evidentiary Procedures, describe the sample handling and transfer systems in detail; this section pertains specifically to the handling of the sample data.

Upon arrival at LAS, environmental samples are logged into the sample management data base. Sample information provided in the system must include:

- Job and client name.
- Date and time of sample collection (to track holding time).
- Date of sample arrival at the laboratory.
- Client sample ID number.

- Corresponding internal LAS sample ID.
- Types of analyses requested.
- General observations concerning the conditions under which the samples arrived.
- Number and types of samples.
- Designation of samples for quality assurance analysis, if required.
- Storage location.

10.4 SAMPLE BATCHING

Before analysis, samples shall be grouped into analytical batches and ordered according to assigned LAS batch IDs. A distinct sample ID number should be assigned to each QA and QC sample to ensure correct identification and inclusion of the correct number of quality assurance samples in each batch.

The LAS follows all method-specified requirements for sample batch size by sample matrix type and by QC sample type and frequency requirements. Because of specialized client requirements, often due to short holding time requirements or other needs, a smaller than normal ratio of environmental samples to QC samples may be necessary. Although this may not be the most efficient approach for sample throughput, the client will be provided with technically sound, high-quality data. The LAS generally conforms to the EPA CLP "batch" definition of a sample delivery group (SDG), which includes 20 consecutive samples, whether they are received in one day or are cumulatively received over a maximum 14-day period. The 14-day limit is frequently prohibitive if holding time must be considered, especially for volatile and cyanide analyses, and semivolatile and pesticide extractions.

10.5 ANALYTICAL DATA DOCUMENTATION

Accountability for analysis begins with receipt of the samples. At LAS, laboratory personnel typically use bound logbooks with prenumbered pages or sample preparation and analysis bench sheets to record data. Validation of measurement data is easily accomplished by requiring the analyst to review, date, and sign data for each analysis on the day completed. This validation can be further strengthened by providing space for the laboratory supervisor's (or designee) signature, which indicates that he or she has witnessed the data production and the completion of the analysis. Hard-copy data generated by a computer can be permanently affixed in the logbook; hard copy so affixed is acceptable as an original record of sampling and laboratory logging. All original raw data (e.g., chromatograms, logbooks) are maintained in the laboratory while in use, then forwarded to Document Control for long-term storage.

10.5.1 Standards and Reagents Data

The working standards made from certified materials must be labeled with complete information (i.e., standard preparation dates, standard ID, concentration of each constituent (if possible), solvents used, expiration dates, and preparer's name). To ensure standards traceability, this information and further details regarding the preparation of the working standards should be recorded in a bound standards logbook. The documentation should include lot #, concentrations, preparation data, date prepared, storage conditions, preparer's initials, and expiration dates.

10.5.2 Instrument Operation Data

Specific injection/analysis and maintenance logbooks are maintained for each instrument.

These logbooks contain records of all routine and emergency maintenance, tuning, calibration, and analytical activities conducted on the instrument. The project name, the date that the analysis is performed, and the names of the analyst(s) who operated the instrument should be recorded on each page. Upon completion of an operational period, each responsible analyst must validate the information by signing and dating the bottom of the page. Twice each month, each supervisor or designee verifies the accuracy of the information recorded by signing and dating the bottom of the page. Periodically (typically during an internal on-site evaluation) the Quality Assurance Department representative reviews the LAS notebook to ensure that standard procedures are being followed, then verifies the review by initialing and dating each page. The main portion of each page may contain information regarding instrument maintenance and modification, tuning and calibration activities, instrument settings, instrument operating conditions, and the sample analyzed. If automated data management systems are used, reference to the data file for each standard or sample should be recorded.

Hard-copy instrument readouts (such as chromatograms and integrator tapes) must be labeled with analysis date, time, type of analysis, sample ID, and reference to the calibration curve used for quantification; the identification of chromatographic peaks also should be noted. The analytical data package is filed in the Document Control Department.

10.6 MAINTENANCE OF LOGBOOKS

Logbooks are invaluable documentation tools, whether they are used in sample receiving or sample preparation and analysis operations. Regardless of their specific purpose, some general rules apply to the maintenance of LAS logbooks. LAL-90-SOP-0006 details the procedures for logbook maintenance; an overview is provided below.

Logbook entries should be completed in black or blue ballpoint ink. Complete information (e.g., dates, data, sample numbers, observations) should be legibly entered so that an examination by a supervisor, auditor, or another analyst can easily determine what was done, by whom, when, and the results. After the last entry is made, the analyst signs the page. If more than one entry is made on the same page, the analyst should initial and date for each entry. Corrections are made by drawing a single line through the incorrect entry; the line must not obscure the entry. The correct information is then entered and is initialed and dated. The use of correction fluid is prohibited. If the page is not completely filled out, a "Z" or a "slash" should be drawn covering the blank section of the page.

Loose sheets, such as computer printouts or certification information, may be permanently affixed to the logbook provided that the analyst initials and dates over the pasted record. Original pages are never removed from the logbook. The use of bound logbooks encourages a chronological sequence of data insertion. Numbering of pages encourages use of data in sequence, and a table of contents ordered according to date, time, sample ID, type of analysis, type of sample (i.e., routine, blank, and duplicate), and identity of analyst aids in referencing data.

Bound, numbered laboratory logbooks are issued by the Document Control staff. Each logbook is tracked by the Document Control staff, and all completed logbooks are transferred to Document Control for long-term storage if they are not used as a reference document.

10.7 DATA REPORTING FORMATS

The way in which data are reported depends on the specific needs of the client. To meet the special needs of our clients, LAS produces numerous types of "standard" deliverable

packages appropriate for reporting of inorganic, organic, and radionuclide analyses data. The standard packages are suitable for (1) clients who are interested only in determining the concentration of specific constituents of interest; (2) sampling and analysis projects that require analytical data to be presented for review by local and state regulatory agencies; and (3) environmental projects that involve remedial investigations (RIs), feasibility studies (FSs), and site cleanup operations mandated by EPA or by state regulatory authorities. The standard types of data deliverables include data packages that provide CLP-level documentation for inorganics and organics and CLP-like documentation for radiochemical analyses.

General information such as dates of sample collection (if known), sample receipt, sample extraction (if applicable), and analysis are also provided on the data reporting forms for every data package.

Data in electronic format are also provided as required by the clients.

10.8 DATA MANAGEMENT SYSTEM

Large portions of the analytical data generated in laboratories are initially recorded on bench sheets, then transferred onto data reporting forms or into computer data bases, and the danger of data transfer error increases each time the data are copied. To minimize such errors, at LAS every effort is made to enter data directly into the computer data base, or direct output of data is provided from instruments (e.g., AAS, ICP-AES, GC, GC-MS) into a data base.

At LAS, the Laboratory Data Management System (LDMS) serves as the central repository for sample data. Five subsystems are integrated into the LDMS: Sample Management, Test Scheduling, Sample Preparations Specification, Quality Control Charts, and Disposal. These subsystems are combined using the Oracle Relational Data Base Management System. The LDMS provides each sample with a unique bar-code identifier which links sample data (e.g., preparation data, analysis data, result data, turnaround times) to the customer or SDG. This system of barcodes allows the samples to be tracked throughout the laboratory, providing location, status, and time constraints. The LDMS also provides an excellent audit trail, capturing such information as the name of the analyst who made the change (user ID and password), the reason for the change (the analyst is required to input the reason), the data existing before the change, and the changed data. This audit trail is always accessible for review by management and the Quality Assurance staff only. Reports in the LDMS are generated in standard or tabular formats to meet the customer's specific needs.

Computer security is controlled by a User Name/Password System at three levels: Local Area Network, Unix Operating System, and the LDMS. Security access to the subsystems is restricted to only those personnel allowed to add, modify, and change data. Other personnel within the LAS can be defined as "view only." This status entitles these users to examine status and result data, but not to change the data. The Audit Module monitors and tracks access activity and requires electronic comments to be entered should results be modified.

Testing of software and hardware at LAS is initiated through request forms: Change Request, New Requirements, and Discrepancy Reports.

Software QA. LAS employs LDMS software purchased from an external source. Project definition, functional design, and implementation phases of the LDMS software is required to be implemented and documented adequately.

All internally developed software related to data generation activities is evaluated by Computer Center staff, the Operations staff, and the independent QA Department staff to ensure that computer-generated data are accurate and meet the end users' specific requirements. The software review is documented on the Lockheed Software Verification Form presented in Figure 3 and requires the approval of the independent QA Department staff. The LAL-91-SOP-123 and LAL-91-SOP-124 delineate LAS-specific procedures related to the independent validation, verification, and documentation of the software according to its intended use.

10.9 DOCUMENT CONTROL

LAS is equipped with a centralized document control facility managed through the Document Control Section. Document Control provides a secure location to store and account for all official LAS records. The Document Control Section is responsible for:

- Storing all applicable client data, including sample analysis data, QC data, and the final delivery report.

- Storing, maintaining, and managing records for LAS SOPs, quality assurance data files, laboratory logbooks, laboratory certifications, performance evaluation reports, health and safety records, administrative operations, electronic media data, and facility security data.

- Maintaining and monitoring off-site facilities for long-term storage and archiving of all records described above.

The location of each document control item is given in the Document Control Inventory Index Notebook. A document control number, following a standard numbering convention, is assigned to every accountable item in the Document Control system.

Quality assurance files in hardcopy and/or electronic media include: internal and external PE Studies; corrective action reports; federal and state certifications; internal and external systems audit reports; certificates of internal PE materials, standard reference materials, and support equipment; MDL study data; calibration and internal QC data; control charts; annual balance/weight certifications by external vendors; sample preparation, analysis, and maintenance logbooks; training data; and project-specific QA/QC information.

Access to the Document Control room is restricted. Only Document Control Section and designated technical leaders and management personnel have access to the document control files. Upon request, section personnel retrieve file item(s). A work table is available to laboratory personnel for reviewing requested items. When an item is removed, it is recorded on a sign-in/sign-out sheet.

All records of chemical analyses, including all raw data, calculations, quality control data, and reports, are kept for a minimum of three years unless otherwise specified by the customers.

LAL-90-SOP-0001 details the procedures outlined above and specifies document control for sample analysis data, SOPs, QA and QC data, LAS administrative files, and electronic media.

Figure 3. Lockheed Software Verification Form

LOCKHEED SOFTWARE VERIFICATION FORM

SOFTWARE TITLE			DATE	NUMBER
DEVELOPER	DEPT CODE	PHONE	TYPE OF SOFTWARE (MACRO, PROGRAM, ...)	VERSION.RELEASE
OPERATING HARDWARE	SOFTWARE DEVELOPED IN (LOTUS, EXCEL, DBASE ...)	MANUFACTURED ☐ IN-HOUSE ☐	MANUFACTURER	

DESCRIPTION OF SOFTWARE/FUNCTION

DESCRIPTION OF TEST PLAN (FUNCTIONAL TESTS, RANGE TESTS, PERFORMANCE TESTS, CALCULATION TESTS, INPUTS, OUTPUTS, ...)

TESTER(S) ASSIGNED/DATE

ASSESSMENT OF TEST RESULTS (FUNCTIONAL, RANGE, PERFORMANCE, CALCULATIONS, INPUTS, OUTPUTS, SITE ANY DEFICIENCIES, ...)

OTHER COMMENTS (BE CAREFUL)

TESTER(S) SIGNATURE/DATE

☐ APPROVED FOR IMPLEMENTATION.

☐ ADDITIONAL DEVELOPMENT OR ANALYSIS REQUIRED. ASSIGNED TO: _____ By: _____

(DESCRIBE UNDER COMMENTS SECTION.)

☐ DISAPPROVED. (EXPLAIN UNDER COMMENTS SECTION.)

COMMENTS SECTION

TESTER/SUPERVISOR SIGNATURE/DATE

☐ SOFTWARE VERIFICATION ACCEPTED BY QA.
REQUEST CLOSED.

QA SIGNATURE/DATE

CHAPTER 11
DATA EVALUATION AND VALIDATION

11.1 INTRODUCTION

Data validation is the process in which data are assessed for acceptability on the basis of established criteria. The primary objective of this quality assurance function is to assess and document the technical quality of the data generated from inorganic, organic, and radionuclide determinations performed by the laboratory analysts. A supporting objective is to evaluate the overall performance of the measurement processes on a continuous basis. The LAS staff do not interpret data useability for a client. We assess and document the technical quality of data in order to help the client evaluate the useability of the data and make sound environmental decisions.

The LAS Quality Assurance Department personnel use a structured mechanism for validating analytical data, thereby minimizing subjectivity. This chapter provides an overview of the procedures used in reviewing and validating analytical data generated by the laboratory staff. LAL-90-SOP-0008, LAL-90-SOP-0012, LAL-90-SOP-0013, and LAL-91-SOP-0088 describe in detail the procedures and the acceptance limits involved in reviewing and validating data. LAL-93-SOP-0274 delineates the procedures for assessing data integrity using electronic media. These SOPs are also useful in training Quality Assurance and technical staff to validate and review data correctly and consistently.

The data validation procedures described here comply with the requirements specified in:

- U.S. EPA Laboratory Data Validation Functional Guidelines for Evaluating Inorganic, Organic, and Pesticide/PCB Analyses (1988).
- U.S. EPA National Functional Guidelines for Organic Data Review (1991).
- U.S. EPA Guidelines and Specifications for Preparing Quality Assurance Project Plans (QAMS-005/80).
- U.S. EPA Contract Laboratory Program Statement of Work (current version).

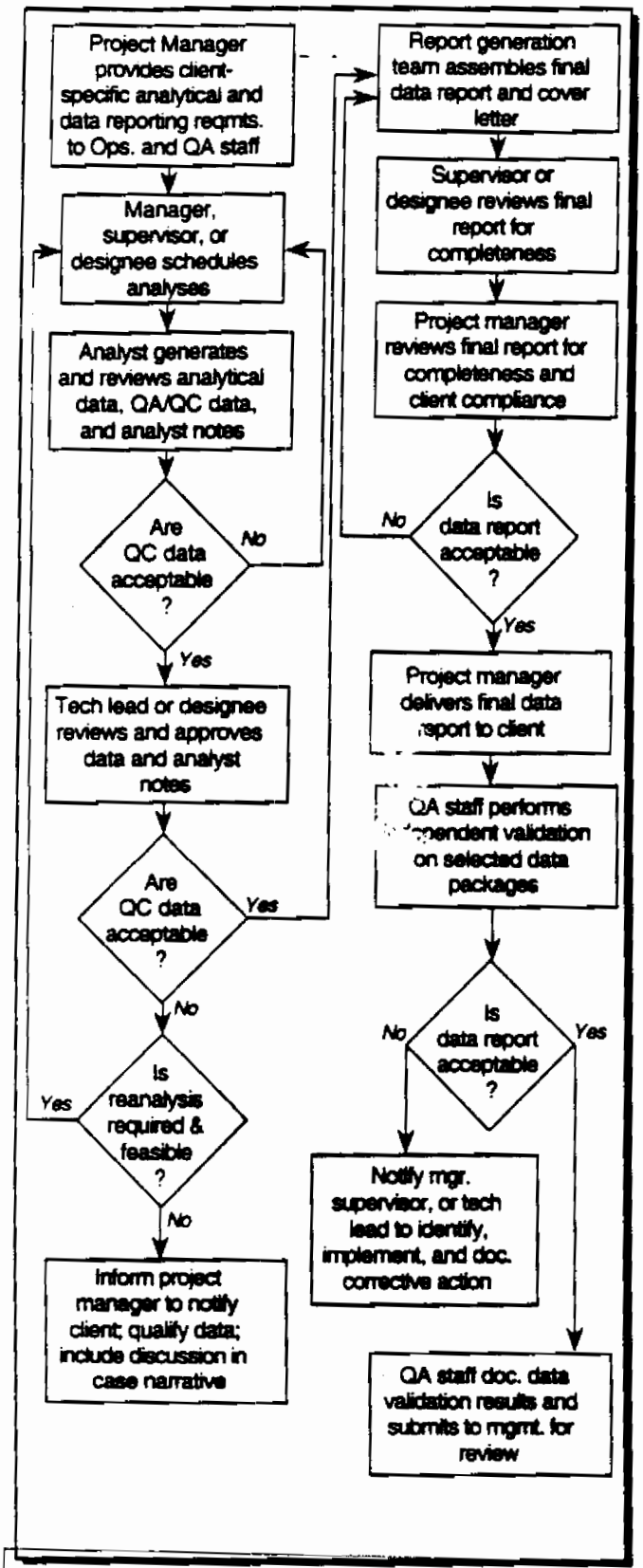
11.2 TECHNICAL DATA REVIEW

To ensure the identification and correction of potential anomalies early in the data generation process, all measurement data produced during laboratory analysis are reviewed, first by the analyst, then by the supervisor or a designated technical specialist. Quality Assurance personnel review selected data packages in depth to further ensure data integrity. Figure 4 is an overview of the LAS data review flow. LAL-90-SOP-0008 describes the technical review of data packages.

11.2.1 Data Review By Analyst

During the course of the analytical run, the analyst incorporates all applicable QC check samples as specified by the standard method of interest documented in the LAS SOPs. Following each QC sample analysis, the analyst performs necessary calculations either manually

Figure 4. Overview of the LAS Data Review Process



or using appropriate software. If a QC check exceeds acceptance criteria, an appropriate corrective action (e.g., redigestion/reextraction, dilution, recalibration, reanalysis) is identified and implemented. In the event that QC analyses are not acceptable and an appropriate corrective action cannot be performed, data are qualified using standard data qualifiers. Information related to the analytical run is thoroughly documented in the injection or instrument logbooks. Also, the analyst completes a checklist or a batch narrative to indicate that he or she has reviewed the data.

11.2.2 Data Review by Section Supervisor or Technical Specialist

Following completion of a batch of samples, the section supervisor or designated technical specialist reviews the data package (generated by the analyst in either electronic or hard-copy format) to ensure that the calculations are accurate, that internal QC samples are analyzed at the required frequency, and that QC data meet method- or LAS-established criteria. If discrepancies are identified, the supervisor discusses and resolves them with the analyst by assigning an appropriate corrective action, which may include recalibration, repreparation, and reanalysis of samples in question. The Supervisor or designee also performs a completeness review of the data report before it is forwarded to the Project Manager. The reviewer documents his or her review on the checklist, the data reporting forms, or the raw data.

11.2.3 Data Review by Project Manager

The final review is performed by the Project Manager (i.e., client services representative) who evaluates the data package for completeness, accuracy, consistency, and client compliance. A data package that meets all the requirements is then submitted to the client. The final data package is also submitted to

Document Control for long-term storage to ensure data custody.

11.2.4 Data Review by Quality Assurance Department Staff

For selected data packages, the Quality Assurance Department staff performs independent, extensive evaluation of data quality, report completeness, and client compliance to identify systematic problems associated with the data production process. LAL-90-SOP-0012, LAL-90-SOP-0013, and LAL-91-SOP-0088 describe the procedures for inorganic, organic, and radionuclide data validation, respectively. LAL-93-SOP-0274 describes the procedures for assessing data integrity using electronic media (tape audits) for analyses that employ GC and GC/MS techniques.

A preliminary review of the chain-of-custody record and Sample Discrepancy Report for sample-specific information (e.g., sample collection, preservation, holding time requirements, cooler/sample condition upon arrival) is performed to assess sample integrity. Holding times between sample collection and analysis are checked to assess potential degradation or loss of analytes of interest.

Analytical precision and accuracy are evaluated by using QC check standards, LCSs, surrogate spikes, matrix spikes, matrix spike duplicates, and unspiked duplicates and tracer recovery to estimate the degree of variance around the reported value and any bias effect due to matrix or laboratory sample processing procedures. In addition, data obtained from external SRMs, submitted as unknowns (blind) samples to the analyst, are evaluated to assess the accuracy of the overall laboratory system and the reliability of the data. Potential for background contamination that may result from sample containers, reagent water, reagents or solvents used during digestion or extraction, cross contamination during storage, or carryover during analysis are evaluated using instrument (calibration), method, and holding blank data. MDLs and RDLs are evaluated to ensure that minimum detectability requirements specified by the method or client are met.

Random errors resulting from incorrect calculations, transcriptions, unit conversions, and switched samples are also examined by independent recalculations against the raw data.

If systematic errors are identified during the QA data review, a Nonconformance & Corrective Action Record (NCAR, Figure 5) is initiated by the reviewer and submitted to the responsible LAS staff. The corrective action implemented is then documented on the NCAR by the responsible party and verified by the QA staff. Collectively, the evaluation comments regarding the data quality are documented in a checklist, which is specific to a client's batch of samples or to an analytical batch, to indicate whether DQOs are met and that resultant data are valid. A detail description of the corrective action program is provided in LAL-92-SOP-0190 and in Chapter 15.0.

Figure 5. LAS Nonconformance & Corrective Action Record

NONCONFORMANCE & CORRECTIVE ACTION RECORD

NCAR -

Page 1 of

Nonconformance

___ Analytical Method	___ Sample Handling & Management	___ Communication
___ Analytical System	___ Data Review & Validation	___ External PE Studies
___ Sample Preparation	___ Data Reporting	___ Other *(describe below)*
___ Standard Prep & Traceability		

Detailed Description of Nonconformance

Project Name/Job Name	Method	Prep Batch ID	Analysis Batch ID
Originator's Signature	Date	Supervisor's Signature	Date

Corrective Action

Detailed Description of Corrective Actions Implemented or Planned

Responsible Person's Signature	Date
Supervisor's Signature	Date
Client Notification Required? ___ yes ___ no ___ n/a	Date Contacted

Corrective Action Verification

Detailed Description

	Date

CHAPTER 12
ASSESSMENT OF ANALYTICAL DATA QUALITY

12.1 INTRODUCTION

Data corresponding to four of the six primary analytical DQOs (see Chapter 4) can be assessed quantitatively. Quantitative assessment of precision, accuracy (as bias), and completeness is discussed here. Detectability, which must be assessed during the analytical process, is discussed in Chapters 4, 7, 8, and 9.

12.2 PRECISION

Precision is an estimate of variability. It is a measure of the agreement among individual measurements of the same sample or same concentration (e.g., performance evaluation samples, matrix spikes and matrix spike duplicates, or unspiked duplicates). For analytical measurements performed at LAS, precision is expressed as intralaboratory precision (precision within a single laboratory).

Intralaboratory precision estimated from field duplicate data represents variability that results from sample collection, processing, analysis, and inhomogeneity. On the other hand, analytical laboratory duplicate data (i.e., spiked and unspiked duplicates and LCS duplicates, if applicable) represent variability that results from the measurement process (i.e., sample processing and analysis in the laboratory). Further, precision within a single laboratory can be evaluated in terms of repeatability (within-run precision) and reproducibility (between-run precision).

Interlaboratory precision can be best estimated through repeated measurements of the same sample type at the same concentration. These samples (external performance evaluation audit samples) are used to establish overall analytical laboratory performance.

Precision for a duplicate pair (a sample and its duplicate) is calculated as RPD or as RSD when more than two data points are involved. For radiochemistry, RER is also used to determine precision. (See Section 4.1 for further explanation of the RPD, RSD, and RER equations.) Large percent RPD/RSD indicates poor precision between the sample and its duplicate for a given constituent. Ideally, values for a sample and its duplicate would be equal, and the standard deviation would be zero. However, as the mean concentration of the duplicate pair approaches the detection limit for that measurement, a high RPD or RSD is expected because large relative errors may occur at low concentrations. Consequently, the precision limits given in Appendix A represent precision at approximately 5 times the MDL, a level at which the precision is expected to stabilize.

12.3 ACCURACY (BIAS)

Accuracy (bias) is determined by analyzing a reference material of known constituent concentration or by analyzing a sample to which a known concentration or amount of constituent has been added (i.e., LCS, matrix spike, surrogate spike samples, and yield tracer). The accuracy estimate may apply only to a specific portion of the measurement system rather than to the entire measurement system. Bias can be caused by the sample matrix, sample preparation procedures, analytical method, measurement system, and improper sample handling practices. Accuracy is calculated as a percent recovery or as a percent bias. (See

Section 4.1 for further explanation of the accuracy equations.)

Analytical bias can be determined through the analysis of a standard reference material (e.g., QCCS, ICV, CCV) or a certified performance evaluation audit sample which has a known concentration. Bias resulting from sample matrix is determined through the matrix spike analysis. Sample-handling accuracy is estimated through the analysis of matrix spikes and of LCSs that undergo a digestion or extraction process performed on each sample matrix associated with a group of 20 or fewer samples.

12.4 COMPLETENESS

Completeness of data collected can be compared directly to the DQO (95%). An environmental project can produce 100% data completeness; however, the results may not be representative of the constituent concentrations actually present. For example, the analytical method might be biased, or the sampling frequency or locations may not provide a representative indication of the actual distribution of the constituent in the matrix sampled.

CHAPTER 13
INTERNAL SAMPLE CHAIN-OF-CUSTODY AND EVIDENTIARY PROCEDURES

13.1 INTRODUCTION

The LAS chain-of-custody procedures are applicable to all samples received by the LAS or its subcontractors, regardless of sample origin or disposition. All environmental samples received are considered physical evidence and must be handled in accordance with certain procedural safeguards. The LAS chain-of-custody program, through careful documentation, ensures traceability of the handling and possession of each sample from time of receipt through completion of analysis and data reporting. The program is in compliance with procedures established by the NEIC Contract Evidence Audit Team for evidentiary handling of samples in the CLP.

13.2 RESPONSIBILITIES

The primary responsibility for the maintenance of the chain-of-custody records belongs to the Sample Custodian; however, all LAS personnel are responsible for maintaining the integrity and traceability of samples that are assigned to them, in accordance with LAL-90-SOP-0009. Furthermore, the Operations Manager, Quality Assurance Manager, and each supervisor must ensure that all personnel are familiar with and follow the LAS chain-of-custody policy and procedures.

13.2.1 Sample Custodian

The Sample Custodian has the primary responsibility for receiving and accurately logging samples into LDMS as detailed in LAL-90-SOP-0002. Samples and standards containing radioactive constituents are received, logged in, and transferred according to procedures detailed in LAL-91-SOP-0085 and in LAL-91-SOP-0113. In addition to sample receipt, the Sample Custodian must fully document sample custody and communicate information to the appropriate parties. Other custody duties include:

- Radiation screening of all coolers and samples.
- Checking sample containers for breakage and leakage.
- Placing samples in appropriate and secure storage areas.
- Controlling access to samples in storage and ensuring that the chain-of-custody SOPs are followed when samples are removed from and returned to storage.
- Maintaining sample identification files, including documentation for any missing or disposed samples.
- Ensuring that conditions of storage facilities are properly monitored and maintained (e.g., refrigerator temperatures) located in the sample receiving area.
- Cleaning LAS shipping containers.
- Returning shipping containers to the client's field operations team(s).
- Notifying the Client Services Department

representatives of issues related to cooler and sample condition and any discrepancies upon receipt.

Sample Custodian alternates are also available as needed. The alternates are fully trained to perform the duties of the Sample Custodian and are proficient in all aspects of sample shipping, receiving, and tracking. The alternates also assist the Sample Custodian during peak activity periods.

13.2.2 Analysts

All analysts and technicians handling samples must follow the chain-of-custody protocols described in LAL-90-SOP-0009. These procedures include:

- Maintaining sample integrity, which may involve refrigeration, prevention of light exposure, or protection from dust, moisture, or other forms of contamination.
- Maintaining accurate, up-to-date logbooks and records (LAL-90-SOP-0006).
- Updating sample status in LDMS.
- Communicating pertinent information to applicable parties.
- Returning extracts, digests, and samples to the proper location upon completion of processing or analysis.

13.3 CHAIN-OF-CUSTODY ELEMENTS

13.3.1 Sample Labels

At LAS, field samples are typically received with labels affixed at the sampling site in order to prevent misidentification of samples. The information on the label generally include:

- Sampling location
- Client sample ID
- Date and time of sampling
- Constituents of interest (if space permits)
- Name of sample collector
- Chemical preservative(s) used
- Other relevant information

Figure 6 shows the internal chain-of-custody sample seal and label. Each sample received at LAS has affixed to it an internal label that includes a unique LAS sample ID, the contract number, the sample matrix, and the required methods of analysis.

13.3.2 Sample Seal

When samples are shipped to LAS by common carrier (e.g., air freight), the shipping container and individual sample bottles should be sealed to ensure the integrity of samples during transportation. The sample seal may contain the date of sample collection and the client's sample ID numbers as per project requirements.

13.3.3 Chain-of-Custody Form

In order to establish the documentation necessary to trace sample possession from the time of collection, a serially numbered chain-of-custody form should be completed and should accompany every sample. Figure 7 is the LAS chain-of-custody form to be used for environmental analyses. This form must contain the following types of information:

- Client name and address
- Sample identification

Figure 6. LAS Internal Chain-of-Custody Sample Seal and Label

Lockheed Analytical Laboratory 1-800-582-7605	CUSTODY SEAL Sample ID: _____ Signature: _____ Date: _____

- Signature of sample collector
- Date and time of sample collection
- Sample type (e.g., ground water, soil)
- Location description of sampling site
- Number of containers
- Chemical and physical constituents and methods for which analysis will be conducted
- Preservatives
- Signature(s) of person(s) involved in the chain of possession
- Inclusive dates and times of possession
- Internal temperature of shipping container (cooler chest) upon arrival at the laboratory
- Condition of samples upon arrival at the laboratory
- Cooler and sample survey for radioactivity

13.3.4 Log-in Chain-Of-Custody Report

The sample log-in data sheet generated by the Sample Custodian must accompany the sample(s) on delivery to the individual analytical laboratory and should clearly identify which sample containers have been designated for each required chemical and physical constituent. This form should include the following types of information:

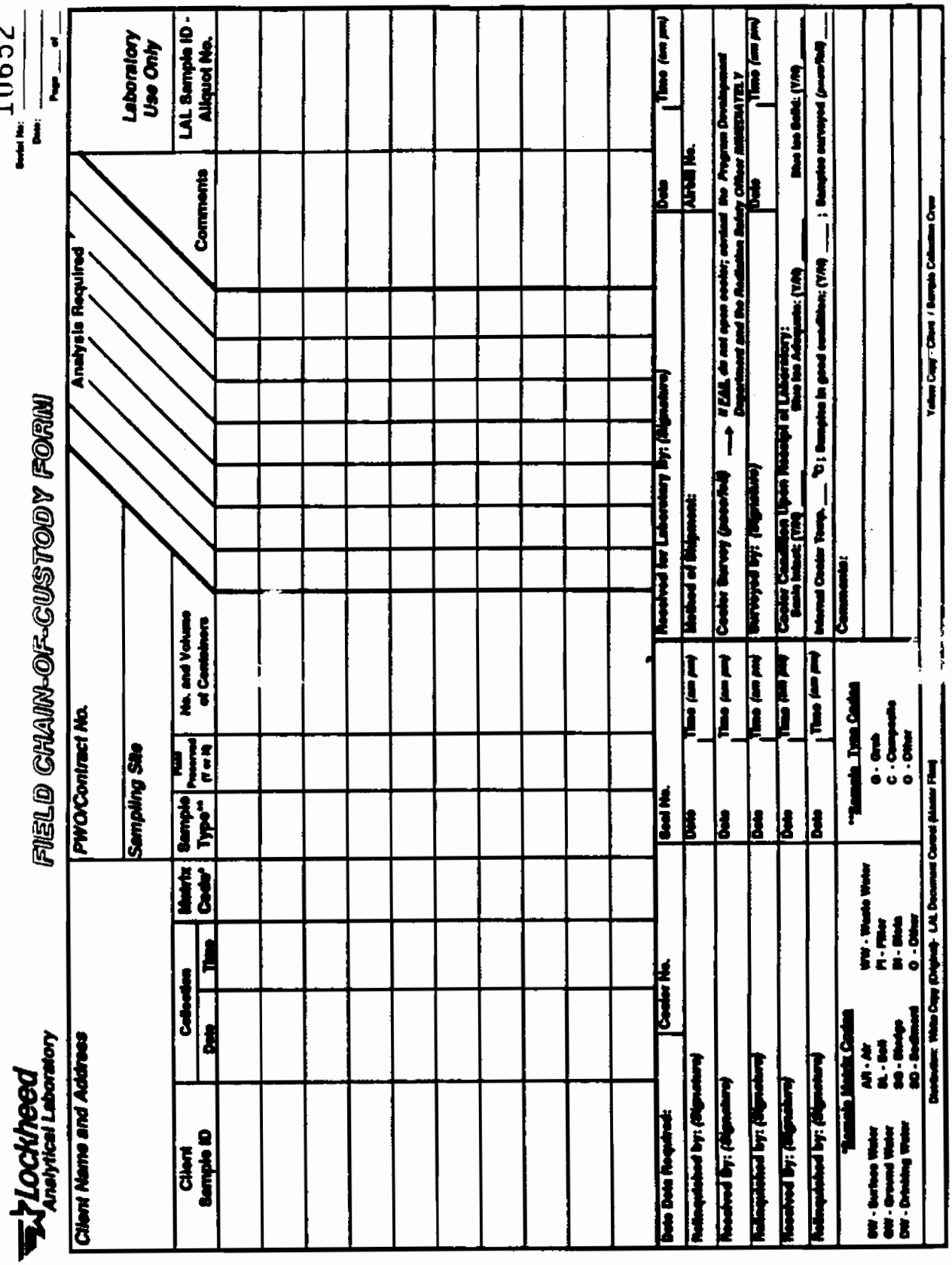
- Name of person receiving the sample (Sample Custodian)
- Client and LAS sample ID numbers
- Date of sample receipt
- Analyses to be performed
- Cooler and sample condition
- Date sample collected (to trace holding time requirements).

13.3.5 Sample Receiving Checklist

The Sample Receiving Checklist generated by the Sample Custodian must accompany the sample(s) on delivery to the individual analytical laboratory and should clearly identify cooler/sample condition upon receipt and any discrepancies identified during sample log in. This form should include the following types of information:

- Cooler condition (i.e., presence of custody seals, chain-of-custody form, and sufficient coolant material and radioactivity survey).
- Sample condition (i.e., bottle labeling, proper containers types, preservation, sample volume, headspace for VOA vials).

Figure 7. LAS Chain-of-Custody Form



- Miscellaneous items (e.g., identification of samples requiring short holding times and samples to be subcontracted).

13.3.6 Analysis Records

After the environmental sample has been received in the laboratory, the Sample Custodian or the appropriate laboratory personnel shall clearly document the processing steps that are applied to the sample. All sample preparation techniques (e.g., extraction or digestion) and analytical methods used must be documented in the bound logbooks or on bench sheets. Experimental conditions, such as the use of specific reagents (e.g., solvents or acids), temperatures, sample pH, and instrument settings, should be noted. The results of the analysis of all QC samples should be recorded if generated using manual instrumentation; these results should be identified in a manner that allows them to be easily associated with the corresponding batch of routine samples. Automated analyses generate electronic format data. The laboratory logbook also should include the date and name of the person who performed each sample processing and analytical step.

All pertinent laboratory information discussed above may be recorded on preprinted forms (e.g., bench sheets) or on computer-generated data reporting forms.

13.4 SAMPLE RECEIPT PROCEDURES

Sample receipt is to be completed only by the Sample Custodian or designated alternates in accordance with LAL-90-SOP-0002 and LAL-91-SOP-0085 for samples potentially containing radionuclides.

13.4.1 Shipping Container Check-In

When the courier has delivered shipping containers (e.g., coolers) to the loading dock, the Sample Custodian (1) checks the number of containers against the airbill or similar form, (2) signs appropriate courier forms, (3) notifies the courier, Operations Manager, Quality Assurance Manager, and the Client Services representatives of any damage to the container(s), and (4) verifies that the chain-of-custody seal on the shipping container is intact. The boxes are then monitored for radiation levels, and if the container is deemed safe, the shipment is then moved to the sample receiving room, where the Sample Custodian or designee (1) records the sender's (e.g., client's) name, date of shipment, and shipping container condition, (2) places the container under the fume hood and removes the chain-of-custody form from the shipping container, (3) inspects the interior of the shipping container, (4) checks the temperature of the shipping container interior, and (5) documents all applicable information on the chain-of-custody form and on the sample receiving checklist. If the sample containers inside the shipping containers are undamaged, the shipping container can be transferred from the fume hood to a work bench for sample check-in.

If samples are determined to be potentially radioactive, the packing is inspected to ensure that it is intact and that it is not leaking. Then sample(s) are moved to the radioactive material receiving laboratory for further handling as specified in LAL-91-SOP-0085. If the shipping container is damaged or suspected of leaking, the Radiation Safety Officer is immediately notified.

13.4.2 Sample Check-In

Once container check-in is completed and documented, the Sample Custodian checks each sample container (e.g., bottle, vial) for leakage or damage, and, if necessary, the container is wiped off and its cap tightened. Broken or damaged containers are documented and set

aside for later disposal. The sample label is then checked against the chain-of-custody form, and, if correct, is entered into the LDMS. An LAS sample ID number is then assigned to the container and entered into the data base, and a bar-coded sample label is affixed to the container. After the contents of the cooler go through this process, the samples are placed on an assigned shelf in a designated refrigerator. Client sample batches are cross-referenced with LAS batch identifiers for tracking purposes.

13.5 SAMPLE STORAGE, TRANSFER, AND DISPOSAL

All samples are stored in locked areas that meet standard storage and preservation criteria for the matrix and analyte. Several refrigerators are designated for storing samples for VOC analyses to prevent cross contamination from other environmental samples. Samples and standards are also maintained separately. Only the Sample Custodian and alternates and Operations Manager have keys to these secured areas. Only the Sample Custodian and alternates are permitted to remove the samples from the storage area; therefore, sample analysis and preparation personnel can obtain samples only with the properly prepared sample batching form and sample tracking form. Once the samples are checked out from the Sample Receiving Area, the LDMS will be sued to track and document the internal chain of custody of all client samples. The new location of each sample will be recorded in the LDMS by Sample Receiving laboratory technicians. This location will be specific and will be a refrigerator within LAS if required. The LDMS will keep a record of all location changes for each sample. This record will be available for retrieval at anytime. If sample containers are changed during processing, the sample preparation analyst prepares new LAS labels, affixes them to the top of the container, and documents this action in the laboratory logbook and on the sample preparation tracking form. Any movement of sample between analysis laboratories or to or from refrigerators will be recorded in the LDMS by the analyst involved. Analysts are instructed to refer to appropriate SOPs for method-specific sample handling and storage requirements. All samples remaining in analysis laboratories awaiting further work will be stored as required. Samples not requiring refrigeration will be stored in designated secure areas in each laboratory. When processing or analysis is complete, the analyst returns the unused portion of the sample and the respective sample tracking form to the Sample Custodian, who then changes the location of the samples in the LDMS data base.

At the discretion of the Sample Custodian, and when storage space is at a premium, samples for which all analyses and data reports have been completed may be transferred to an off-site location for storage and archival for later disposal or, if requested, they may be returned to the client. It is the policy of LAS to dispose of samples 60 days after the submittal of the report to the client unless otherwise specified in writing by the client or as required by regulations and licenses governing laboratory operations. All transfers will be documented (1) on the sample tracking form, which is stored in the Document Control files after the sample analysis and archiving and the data reporting activities have been completed and (2) through the LDMS.

CHAPTER 14
PREVENTIVE MAINTENANCE AND REPAIR

14.1 INTRODUCTION

Preventive maintenance and instrument and equipment repair responsibilities are coordinated through the LESAT Environmental Sciences and Technologies Division Instrumentation Maintenance and Repair Section. It is the responsibility of the engineers in this section to perform maintenance and repairs in coordination with the instrument operators and in accordance with the standard formats and procedures described in LAL-90-SOP-0188 and LAL-90-SOP-0015. Minor instrument and equipment maintenance is also performed by the analysts and other laboratory staff as required.

14.2 PREVENTIVE MAINTENANCE

Preventive maintenance is the scheduled routine action taken to help ensure the proper operation of instrumental systems. A proper preventative maintenance program consists of, but is not limited to (1) the periodic calibrating, tuning, and cleaning of instruments, (2) the periodic changing of oils and filters, and (3) the monitoring of known areas of wear or degradation to ensure the timely replacement of worn parts or components. The criteria used to determine the scope and frequency of preventative maintenance are (1) the instrument manufacturer's recommendations, (2) compiled maintenance data, (3) past experience of the instrument operators, and (4) the maintenance engineers.

In general, preventive maintenance is scheduled quarterly as the instrumental systems are available, even if there is no indication of a negative effect on data quality resulting from the system performance. Personal computers are serviced every 6 months. A preventative maintenance schedule for LAS instrumentation is prepared quarterly by the Instrumentation Maintenance and Repair Section and is distributed to all laboratory departments. The engineer assigned to perform the preventive maintenance on a particular instrument contacts the instrument operator at least one working day before the scheduled maintenance in order to confirm the schedule with the operator. Occasionally, project (e.g., client) priorities conflict with this schedule. It is the responsibility of the instrument operator or section supervisor to reschedule the servicing at an earlier or later date. This practice ensures minimizing instrument "down time" that could affect sample holding times and project deliverable deadlines. All preventive maintenance work is documented on a work order request form prepared by the engineer and signed by the operator or requestor. A carbon copy of the form is retained in the laboratory files and is also documented in the instrument operations logbook.

In addition to the routine maintenance performed quarterly, each instrument operator performs maintenance as needed. This work may include cleaning or replacing analytical columns, injection ports, or transfer lines.

14.3 REPAIRS

Repairs are defined as any unscheduled service or maintenance required on equipment and instrumentation. This work is performed expressly at the request of the operator or supervisor and can include repairs of nonfunctioning instrumentation or of functioning instrumentation not performing optimally.

Requests for repairs are first entered into the service logbook. The engineer assigned to each request then makes a preliminary evaluation of the work required and initiates a work order request form. The work may include such activities as diagnosis, parts procurement and installation, or inventory control. Upon completion of the repair, the work order request form is completed by the engineer and signed by the requestor. A carbon copy of the form is retained in the laboratory files; the work is also documented in the instrument operations logbook.

14.4 MAINTENANCE OF LABORATORY SUPPORT EQUIPMENT

Important aspects of maintaining sample integrity, ensuring precise and accurate sample measurements, and providing worker safety include the proper maintenance, calibration, and inspection of a wide array of support equipment used for sample storage and preparation. This support equipment includes analytical balances, micropipets, thermometers, balances, weights, refrigerators, freezers, ovens, reagent water systems, waste water discharge monitors, fume hoods, ventilation systems, and radiation survey detectors. Routine and periodic checks and maintenance of the support equipment are described in detail in LAL-90-SOP-0015, and a summary is provided below. Maintenance of major analytical equipment, such as GC-MS, AAS, and ICP-AES, is discussed in LAL-90-SOP-0188. Routine checks and maintenance of fire protection equipment, high efficiency particular air (HEPA) filters, hoists, perchloric acid scrubbers, safety equipment (i.e., emergency light, eye wash, safety shower and ground fault circuit), and waste water discharge monitors are also discussed in detail in LAL-90-SOP-0015.

14.4.1 Refrigerators and Freezers

Daily refrigerator and freezer temperatures are recorded in a temperature logbook. The temperature log includes date, time, temperature, corrective action (if required), and initials. Refrigerator temperatures should be at 4 °C ± 2 °C and freezer temperatures at -20 °C to -10 °C, measured with NIST-traceable thermometers. If the units deviate by more than the specified temperature tolerances, adjustments are made to bring them within specifications.

14.4.2 Ovens

The temperature of each oven is measured before use at the dial setting appropriate for the method of determination using NIST-traceable thermometers. The temperature readings are recorded on the Oven Temperature Log assigned to each oven.

14.4.3 Analytical Balances

The accuracy of all analytical balances is checked using "NBS Class S" or "ASTM Class 1" NIST-traceable weights. Balances used daily are checked daily before use. Balances used infrequently are checked before use. The balance accuracy checks are recorded in the logbook designated for each balance. Each analytical balance is certified annually by an independent vendor, and the certification is documented by a label affixed to the balance and on certification forms maintained by Document Control. Details of analytical balance check procedures are provided in LAL-90-SOP-0046.

14.4.4 NIST Class "S" Weight Calibration

On an annual basis, one set of Class "S" weights is sent to an external, qualified vendor for independent calibration. The other Class "S" weights or equivalent used to check the accuracy of balances at LAS are verified against the externally calibrated Class "S" weight set each year. Copies of calibration certificates and the verification data are maintained with the weights and in Document Control files.

14.4.5 Micropipets

Each micropipet is calibrated monthly at three frequently used volume settings in accordance with procedures described in LAL-91-SOP-0175 and in LAL-93-SOP-0205 for inorganic and radionuclide determinations, respectively. Each measurement must be within 1 percent for volumes $\geq$ 100 μL, and 2 percent for volumes $<$ 100 μL. If the QC limit is exceeded, the micropipet is adjusted according to the SOP specifications. The micropipets should be labeled appropriately to indicate the status of calibration. The micropipet calibration checks, calculations, and required adjustments are recorded in a bound logbook.

14.4.6 Reagent Water System

Each day, the resistivity of reagent water for each Nanopure reagent water system is measured and recorded on the form posted on the system. The resistivity for ASTM Type II reagent-grade water must be greater than 1.0 MΩ.cm at 25 °C. If the resistivity drops below 3.0 MΩ.cm, the system's filters are replaced. In general, although directly related to the frequency of use of the system and the feed water condition, prefilters are changed periodically by the LAS maintenance staff. The quality of the reagent water is further monitored continuously through the analyses of method (reagent) blanks for inorganic, organic, and radiochemical analyses.

14.4.7 Fume Hoods

Air flow velocities through hoods are checked and recorded every six months. The air handling system is adjusted, if necessary. If adjustments are made, the velocities must be rechecked. A detailed description of this activity is provided in LAL-91-SOP-0099.

14.4.8 Ventilation System

LAS Maintenance personnel check the positive and negative flow of the LAS ventilation system and adjust the flow as needed. Filters are inspected monthly and are cleaned or replaced, if necessary.

14.4.9 Radiation Survey Detectors

All radiation survey detectors are inspected daily, if used, to ensure proper operation, in accordance with the manufacturer's operation manual and LAL-91-SOP-0173. The portable radiation survey equipment shall be sent to an authorized vendor for annual calibration or when the instrument cannot meet QC limit for daily calibration check.

14.4.10 Thermometer Calibration

On an annual basis, an independent thermometer calibration check is performed using an NIST-traceable thermometer, as described in LAL-90-SOP-0015.

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CHAPTER 15
CORRECTIVE ACTIONS AND CONTINGENCY PLANNING

15.1 INTRODUCTION

If, on the basis of internal or external systems or performance audits, sample handling, routine monitoring of laboratory support equipment, or QC sample analysis results, analytical systems fail to meet the established criteria, an appropriate corrective action must be implemented. The Operations Manager, project manager, Quality Assurance Manager, supervisor, and analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected or if the corrective action will impact the project budget or schedule, the action may directly involve the LAS Director.

15.2 CORRECTIVE ACTIONS

Corrective actions are generally of two types, immediate actions and long-term actions.

An immediate action is designed to correct or repair nonconforming instruments and measurement systems. The need for such an action most frequently will be identified by the analyst as a result of calibration checks and other QC sample analyses.

A long-term action is designed to eliminate causes of nonconformance. The need for such actions is identified by quality assurance and QC systems and performance audits. The systematic nonconformances identified during the data generation process and the appropriate corrective measures taken are thoroughly documented on the NCAR. Examples of this type of action include:

- Training and qualification of staff in technical skills or in implementing the Quality Assurance Program.
- Rescheduling of analytical laboratory routine to ensure analysis within allowed holding times.
- Identifying vendors to supply standards of sufficient purity.
- Revising the quality assurance system or replacing personnel, as appropriate.
- SOP revisions.

For either type of corrective action, the sequential steps that compose a closed-loop corrective action system are as follows:

- Define the problem.
- Assign responsibility for investigating the problem.
- Investigate and determine the cause of the problem.
- Determine a corrective action to eliminate the problem.
- Assign and accept responsibility for implementing the corrective action.
- Establish effectiveness of the corrective action and implement the correction.
- Verify that the corrective action has eliminated the problem.

Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be documented properly. On-the-spot actions are used to correct minor problems, such as recalibration, retuning, or a minor repair (e.g., replacement of a minor part) of a malfunctioning instrument or the correction of poor analytical technique being used by an analyst. These occurrences are documented in the appropriate injection, run, or analysis logbooks. Routine instrument maintenance, malfunctions, and power failures are documented in the appropriate instrument maintenance logbooks. The nonconformances systematic in nature are documented and monitored through the NCAR forms. The closed-loop corrective action program is described in detail in LAL-92-SOP-0190. Corrective actions specific to methods are discussed in appropriate SOPs.

15.3 CONTINGENCY PLANNING

A comprehensive Quality Assurance Program must emphasize contingency planning and actions to prevent problems from occurring and to ensure timely, effective completion of a measurement effort. The LAS has contingency plans for the areas listed below. Contingency plans specific to the work areas are also important to the smooth functioning of the LAS and should be addressed and updated periodically by all LAS managers and supervisors.

- *Staffing* - A primary objective is to ensure that qualified staff are always available to perform the necessary analytical work, regardless of employee turnover, vacation, illness, or other absence. The resolution to this issue is to (1) anticipate critical staffing needs and recruit qualified staff to maintain work flow, (2) ensure timely hiring of candidates, and (3) continuously ensure cross-training of existing staff to provide back-up capabilities. Other Lockheed staff who have particular expertise in analyzing difficult samples can also be consulted for advice and problem resolution.

- ***In-house Service Experts*** - Our preventive maintenance program is designed to minimize malfunctions, permit simple adjustments, and ensure fewer and shorter breakdowns of critical analytical equipment. Procedures in place to maximize instrument up-time are described in Chapter 14.

- ***Redundant Instrumentation and Support Equipment*** - In most cases, duplicate instrumentation is available to ensure uninterrupted work flow. It is the responsibility of the section supervisors to ensure that analytical personnel are trained in identifying approved alternative methods of analysis, if necessary. For example, most constituents analyzed by using ICP-AES can also be determined by using furnace or flame AAS and ICP/MS, provided that the required detection limits are attainable. Redundant equipment is available for providing the laboratory reagent water and gases necessary for the analytical instruments. In addition, LAS has procedures in place for leasing major instruments, equipment, and computers within a short time frame, should the situation dictate.

- ***Instrument Service Contracts*** - LAS vendor service contracts ensure that vendors supply 24-hour emergency response. These responses include overnight parts delivery or service-engineer assistance to maintain operating capacity.

- ***Subcontractor Analytical Laboratory*** - To support the laboratory during peak periods or in the event of a critical instrument malfunction, LAS has arranged to use qualified analytical laboratories as subcontractors for short-term backup analytical support. Through an extensive process, LAS QA personnel evaluate, identify, and select qualified analytical laboratories before an analytical contract is awarded. In order to qualify, a subcontractor laboratory must pass this evaluation and, potentially, an on-site inspection.

- ***Uninterruptable Power Supply*** - The Exide Electronics Powerware System 50 Uninterruptable Power Supply (UPS) provides line conditioning and backup power to the LAS HP 9000 845 computer system/server. In case of power failure, the laboratory generator becomes the main source of power. The UPS still provides the conditioning and backup. If, during a power failure, the generator becomes ineffective, the UPS serves as the main power source, providing power for 30 to 60 minutes to the HP 9000 Computer. This contingency plan allows sufficient time for the main computer system to be shut down and data archival. All electronically generated data are stored on the main computer system and on the individual PC hard drives. In the event that the main laboratory computer system fails, the analytical data can be retrieved from the PC hard drives.

15.4 IDENTIFICATION AND CONTROL OF NONCONFORMING ITEMS AND MATERIALS

The LAS personnel must follow procedures to identify, segregate, evaluate, and document nonconforming items or materials to prevent inadvertent installation or use. The section supervisors are responsible for overseeing the identification, segregation, review, disposition, and documentation of nonconforming instruments, equipment, and materials at LAS. This activity is described in detail in LAL-93-SOP-0283.

15.4.1 Identification

Upon identification of nonconforming items (e.g., analytical instruments, support equipment, chemicals, reagents, solvents, etc.), appropriate section supervisors, tech leads, or designees are notified and a legible and easily recognizable identification is used to indicate that the item is not in use. Identification of nonconforming items are typically done by marking, tagging, or other methods that shall not adversely affect the end use of the item. The identification shall be legible and easily recognizable.

If identification of each nonconforming item is not practical, the container, package, or segregated storage area, as appropriate, shall be identified.

The out-of-control condition must be documented in the related logbook, worksheet, maintenance record, Nonconformance and Corrective Action Record (NCAR), or in an internal memo.

15.4.2 Segregation

When practical, nonconforming items shall be segregated from conforming items by placing them in a clearly identified area until proper corrective action is taken or until disposition.

For example, expired standards or chemicals shall be segregated prior to further verification or disposal. The small nonconforming analytical or support equipment, such as balances, thermometers, and pipettors, can be

physically segregated from the routine equipment to avoid possible misuse.

15.4.3 Review

Nonconforming items should be reviewed by appropriate section supervisors, technical leads, or designees to determine whether they can be used as they are or whether they shall be repaired or reclassified.

15.4.4 Disposition

The justification for disposition, such as use-as-is, rework, reject, or repair of nonconforming items must be approved by the appropriate section supervisors or technical leads and documented in the related logbook, worksheet, maintenance record, NCAR, or in an internal memorandum.

Repaired, replacement, or reworked items must be tested to verify that required operational conditions and all the QC specifications can be met before use.

15.5 HANDLING OF CLIENT INQUIRIES

Client inquiries are generally received through the project manager or a member of the Program Development group. Typically, the project manager communicates with the client to ascertain the details of the inquiries, including technical data problems, deliverable issues, turn-around-time problems, etc. Technical and deliverable issues are coordinated by the Project Manager and usually involve input from operations, QAD, and managerial personnel. A formal response to the client is coordinated by the Project Manager, but may on occasion be delivered by a member of the Program Development group.

CHAPTER 16
CONTROL OF PURCHASED ITEMS AND SERVICES

16.1 PROCUREMENT

The procurement of LAS instruments, equipment, chemicals, standards, and services is controlled to ensure compliance with specified requirements. LAS operations, facility, quality assurance and purchasing personnel ensure the adequacy and quality of all contractor-purchased articles, materials, and services. LAS personnel plan and implement procurement quality activities to ensure timely and adequate integration with all other elements of the organization having responsibility for control and performance of subcontractors and suppliers.

At LAS, the Source Selection Process is established to review all competitive procurements when the competition involves an evaluation and comparison of cost or price and other technical factors. The source selection procedures are designed to (1) maximize competition, (2) minimize the complexity of the solicitation, evaluation, and the selection decision, (3) ensure impartial and comprehensive evaluation of all offers, and (4) ensure selection of the source whose offer has the highest degree of quality and whose performance is expected to best meet the solicitation requirements. The findings that result from this process provide permanent procurement file documentation.

LAS maintains a documented receiving inspection system which ensures:

- Procured articles, materials, or services indicate evidence of inspections and tests performed by the supplier in accordance with purchase requirements and are accompanied by required certifications (if necessary).
- Chemical analyses and physical tests are conducted according to the approved analytical protocols.

When an article, material, or service procured by LAS does not conform to applicable specifications or other requirements, it is identified as nonconforming, segregated to the extent practicable, and held for review action. LAS has established a documented, systematic technique for the identification, documentation, and control of nonconformances.

16.2 SELECTION AND QUALIFICATION OF SUBCONTRACTOR ANALYTICAL LABORATORIES

Selecting the analytical laboratory that will provide the best complement of subcontract services for an environmental project is of primary importance. It requires an approach to ensure that all data generated by LAS subcontractors are of known, acceptable, and documented quality and are in compliance with the LAS QA Program- and client-specific requirements.

The LAS has established a policy to perform an in-depth evaluation of a laboratory's capabilities to provide analytical services as a subcontractor before an analytical contract is awarded. The objective is to select laboratories that are capable, technically qualified, credible, and competitive in terms of analytical cost. A detailed description of the specific requirements, procurement, planning, on-site systems

evaluations, and control of supplier nonconformances is provided in LAL-93-SOP-0232.

16.3 MATERIALS PROCUREMENT AND CONTROL

The quality of all materials used in the handling, preparation, analysis, and storage of samples at LAS facilities must be of known and acceptable quality so that the effect of the materials on analytical results can be defined. Reagents, solvents, reagent water, gases, and sample containers, as well as laboratory glassware, vessels, and implements purchased by LAS or prepared internally (e.g., compressed air) shall meet all the requirements that are stated in the particular analytical methods or that are otherwise specified by the client.

Chemical reagents, solvents, and gases are available from a variety of sources and in a variety of purity grades, ranging from technical to ultrapure grades. The constituents measured and the sensitivity and specificity levels of the analysis system are key elements in determining the required purity of these materials. In general, if the analytical method does not specify the grade required, "analytical grade" or higher purity will be used. Procedures for acceptance of laboratory chemicals is addressed in LAL-93-SOP-0284.

16.3.1 Responsibility

The section supervisor is responsible for overseeing and ensuring that (1) suitable grades of materials are specified in requisitions, (2) the materials procured meet the applicable requirements, (3) the appropriate certification or other documentation regarding the materials has been provided and is maintained in the Document Control files, (4) the materials are stored safely and properly, and (5) the materials are removed from use when the shelf-life (or other criteria) is expired or otherwise outdated.

16.3.2 General Materials Requirements

16.3.2.1 Inorganic Analyses. In general, analytical reagent grade reagents and solvents are adequate for inorganic analyses; however, trace metal analyses by atomic absorption and emission spectroscopy shall be spectro-quality. Fuel and oxidant gases may be commercial grade. Compressed air can be commercially supplied (dry grade) or supplied by LAS air compressors, provided that proper pressure and filtration of oil, water, and trace metals are maintained.

16.3.2.2 Organic Constituent Analyses. In general, pesticide grade (i.e., nanograde) is the minimum grade acceptable for materials used in organic analyses. Reference grade standards shall be used as necessary. Some gas chromatography systems require that solvents and standards (and environmental samples, as well) be free of certain compound classes. For example, photoionization detectors require that reagents and solvents be free of sulfur and phosphorus compounds because of their interfering properties.

For sample cleanup procedures, the adsorbent materials (florisil, carbon, silica gel, and alumina) are most commonly used. These materials, as well as all analytical reagents, solvents, and other chemicals must be checked to determine suitability for the analyses.

16.3.2.3 Laboratory Reagent Water. In general, deionized ASTM Type II grade water (or better) is used for dilution of samples in preparation of reagent and standard solutions, and for final rinsing of glassware. The specifications for ASTM Type II grade water for resistivity to be greater than 1.0 M Ω.cm or conductivity less than or equal to 1 μS/cm at

25 °C. Organic-free water is required for VOC analyses; however, when determining trace organics by gas chromatography following solvent extraction, "specialty" water such as HPLC grade water must be used. The quality of the reagent water must be monitored through daily resistivity checks and method blank analyses.

16.3.2.4 Laboratory Containers, Vessels, and Implements. Material composition and volumetric tolerances of containers and other vessels used in the sample storage, preparation, and analysis processes can affect the quality of the analytical data.

Soft glass containers are not recommended for general use, especially for the storage of reagents. Chemically resistant borosilicate glass, such as Pyrex® or Kimax® is generally used unless otherwise specified in the analytical protocols. Plastic vessels, containers, and other apparatuses made of Teflon®, polystyrene, polyethylene, and polypropylene are also desirable when specified. Guidelines for selecting the material composition of laboratory containers are provided in Chapter 4. In addition, implements used in the handling of samples, such as spatulas and spoons, and supplies such as aluminum foil and Parafilm® must meet specifications for the matrices and constituents of interest.

In general, volumetric glassware will be of sufficient accuracy for the analytical reagent volumes measurement. This glassware includes volumetric flasks, volumetric pipets (and static and adjustable micropipettors), and calibrated burets. Less accurate types of glassware, such as graduated cylinders and beakers, are also used for specific applications.

16.3.2.5 Calibration Services. Certain items at LAS are calibrated by independent vendors. Inaccurate calibrations performed by independent vendors could lead to malfunctioning equipment, uncertain data quality, and safety problems. The qualifications of the vendors that provide calibration services will be verified by any or all of the following steps:

- checking references for the vendor;
- checking the vendor's certifications;
- Checking the vendor's quality assurance documentation;
- verifying the vendor's qualifications with the equipment manufacturer;
- checking the accuracy of the calibration with certified standards; and
- reviewing previous experience with the vendor.

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CHAPTER 17
QUALITY ASSURANCE REPORTS TO PROJECT MANAGEMENT

17.1 INTRODUCTION

An effective quality assurance program should include formal and frequent reports to inform management and technical staff of progress in the on-going implementation of the quality assurance plan. At a minimum, the following LAS parties should receive regular updates on project quality assurance status: (1) director, (2) operations manager, (3) program development staff, (4) client services (project) staff, (5) section supervisors, and (6) analysts and other technical staff.

17.2 INTERNAL QUALITY ASSURANCE REPORTS

LAS Quality Assurance personnel develop reports routinely for the LAS staff on the following topics:

- General LAS Quality Assurance Program status (weekly and monthly).
- Long-term control charts (monthly).
- Results of external PE and laboratory intercomparison studies (semiannually).
- Results of external and in-house technical system audits (as reports are received from clients or generated).
- Status of completed and outstanding Nonconformance and Corrective Action Records (weekly).
- Assessment of the blind and nonblind data audit (PE) sample data (as data are produced).
- Laboratory accreditation, licensing, and permitting updates (weekly).
- All significant quality-related problems identified during independent data validation and the corrective action procedures that are recommended (as performed).
- Information regarding QA training, regulatory changes, QA-related issues, and recommendations (as performed).
- Local, state, and federal regulatory information (as updates are received).
- Schedules for external on-site audits (as updated).

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APPENDIX A

ANALYTICAL DATA QUALITY OBJECTIVES FOR PRECISION ACCURACY, AND DETECTABILITY FOR INORGANIC, ORGANIC, AND RADIONUCLIDE CONSTITUENTS

Table A-1. Estimated Reporting Detection Limits and Precision and Accuracy Objectives for Inorganic Constituents (4 Pages)

Constituent	Analytical Method	Estimated Reporting Detection Limit		Precision (RPD)[b]	Accuracy (% Rec)[c]
		Aqueous (mg/L)	Solids[a] (mg/kg)		
Alkalinity - High	310.1	30	300	20	100 ± 25
Alkalinity - Low	310.1	30	300	20	100 ± 25
Aluminum	200.7/6010/CLP	0.20	40	20	100 ± 25
Aluminum	200.8/6020	0.05	10	20	100 ± 25
Ammonia-Nitrogen	350.1	0.05	0.50	20	100 ± 25
Antimony	200.7/6010/CLP	0.06	12	20	100 ± 25
Antimony	204.2/7041/CLP	0.06	0.50	20	100 ± 25
Antimony	200.8/6020	0.005	1.0	20	100 ± 25
Arsenic	200.7/6010/CLP	0.20	40	20	100 ± 25
Arsenic	200.9/7060/CLP	0.01	2.0	20	100 ± 25
Arsenic	200.8/6020	0.01	2.0	20	100 ± 25
Arsenic	TCLP	0.50	100	20	100 ± 25
Barium	200.7/6010/CLP	0.20	40	20	100 ± 25
Barium	200.8/6020	0.05	10	20	100 ± 25
Barium	TCLP	10	2000	20	100 ± 25
Beryllium	200.7/6010/CLP	0.005	1.0	20	100 ± 25
Beryllium	200.8/6020	0.005	1.0	20	100 ± 25
Boron	200.7/6010	0.20	40	20	100 ± 25
Boron	200.8/6020	0.10	20	20	100 ± 25
Bromide	300.0	0.10	1.0	15	100 ± 25
Cadmium	200.7/6010/CLP	0.005	1.0	20	100 ± 25
Cadmium	213.2/7131/CLP	0.005	1.0	20	100 ± 25
Cadmium	200.8/6020	0.005	1.0	20	100 ± 25
Cadmium	TCLP	0.10	20	20	100 ± 25
Cerium	200.8/6020	0.005	1.0	20	100 ± 25
Cesium	200.8/6020	0.005	1.0	20	100 ± 25
Cesium	3500-CS	1.0	200	20	100 ± 25
Chemical Oxygen Demand (COD)	410.4	20	N/A	20	100 ± 25
Chloride	325.2	1.0	N/A	20	100 ± 25
Chloride	300.0	0.020	0.20	15	100 ± 25
Chromium (total)	200.7/6010/CLP	0.50	100	20	100 ± 25
Chromium (total)	200.8/6020	0.010	2.0	20	100 ± 25

Table A-1. Estimated Reporting Detection Limits and Precision and Accuracy Objectives for Inorganic Constituents (4 Pages)

Constituent	Analytical Method	Estimated Reporting Detection Limit		Precision (RPD)[b]	Accuracy (% Rec)[c]
		Aqueous (mg/L)	Solids[a] (mg/kg)		
Chromium (total)	218.2/7191/CLP	0.010	2.0	20	100 ± 25
Chromium (total)	TCLP	0.50	100	20	100 ± 25
Chromium (IV)	7196	0.020	0.20	15	100 ± 15
Cobalt	200.7/6010/CLP	0.050	10	20	100 ± 25
Cobalt	200.8/6020	0.005	1.0	20	100 ± 25
Copper	200.7/6010/CLP	0.025	5.0	20	100 ± 25
Copper	200.8/6020	0.005	1.0	20	100 ± 25
Cyanide	335.2/9010/CLP	0.020	2.0	20	100 ± 25
Fluoride	340.2	0.050	0.50	20	100 ± 25
Hydrazine	LAL-92-SOP-0189	0.0010	0.0010	20	100 ± 25
Iron	200.7/6010/CLP	0.10	20	20	100 ± 25
Iron	200.8/6020	0.050	10	20	100 ± 25
Lead	200.7/6010/CLP	0.10	20	20	100 ± 25
Lead	200.8/6020	0.003	0.60	20	100 ± 25
Lead	239.2/7421/CLP	0.003	0.60	20	100 ± 25
Lead	TCLP	0.50	100	20	100 ± 25
Lithium	200.7/6010	0.10	20	20	100 ± 25
Magnesium	200.7/6010/CLP	2.0	400	20	100 ± 25
Magnesium	200.8/6020	0.50	100	20	100 ± 25
Manganese	200.7/6010/CLP	0.015	3.0	20	100 ± 25
Manganese	200.8/6020	0.015	3.0	20	100 ± 25
Mercury	245.2/7470/7471	0.0002	0.15	20	100 ± 25
Mercury	TCLP	0.020	10	20	100 ± 25
Molybdenum	200.7/6010	0.20	40	20	100 ± 25
Molybdenum	200.8/6020	0.005	1.0	20	100 ± 25
Monomethylhydrazine	LAL-92-SOP-0189	0.0010	0.0010	20	100 ± 25
Nickel	200.7/6010/CLP	0.040	8.0	20	100 ± 25
Nickel	200.8/6020	0.005	1.0	20	100 ± 25
Nitrate as Nitrogen	300.0	0.020	0.20	15	100 ± 25
Nitrate/Nitrite as Nitrogen	353.2	0.050	0.50	20	100 ± 25

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Constituent	Analytical Method	Estimated Reporting Detection Limit		Precision (RPD)[b]	Accuracy (% Rec)[c]
		Aqueous (mg/L)	Solids[a] (mg/kg)		
Nitrite as Nitrogen	300.0	0.010	0.10	15	100 ± 25
Ortho-Phosphate as Phosphorus	365.2	0.030	0.30	20	100 ± 25
Osmium	200.7/6010	0.10	40	20	100 ± 25
Phosphorus	200.7/6010	0.050	10	20	100 ± 25
Potassium	200.7/6010/CLP	2.0	400	20	100 ± 25
Potassium	200.8/6020	0.50	100	20	100 ± 25
Selenium	200.7/6010/CLP	0.30	60	20	100 ± 25
Selenium	200.8/6020	0.005	1.0	20	100 ± 25
Selenium	270.2/7740/CLP	0.005	1.0	20	100 ± 25
Selenium	TCLP	0.10	20	20	100 ± 25
Silica	370.1	1.0	N/A	20	100 ± 25
Silicon	200.7/6010	0.10	20	20	100 ± 25
Silicon	200.8/6020	0.10	20	20	100 ± 25
Silver	200.7/6010/CLP	0.10	2.0	20	100 ± 25
Silver	200.8/6020	0.005	1.0	20	100 ± 25
Silver	200.9	0.010	2.0	20	100 ± 25
Silver	TCLP	0.50	100	20	100 ± 25
Sodium	200.7/6010/CLP	2.0	400	20	100 ± 25
Sodium	200.8/6020	0.50	100	20	100 ± 25
Sulfate	300.0	0.10	1.0	15	100 ± 25
Sulfate	375.4/9038	5.0	50	20	100 ± 25
Sulfide	9030	3.0	N/A	20	100 ± 25
Strontium	200.7/6010	0.10	20	20	100 ± 25
Strontium	200.8/6020	0.005	1.0	20	100 ± 25
Thallium	200.7/6010/CLP	0.50	100	20	100 ± 25
Thallium	200.8/6020	0.005	1.0	20	100 ± 25
Thallium	279.2/7841/CLP	0.010	2.0	20	100 ± 25
Tin	200.7/6010	0.20	40	20	100 ± 25
Tin	200.8/6020	0.005	1.0	20	100 ± 25
Titanium	200.7/6010	0.10	20	20	100 ± 25
Titanium	200.8/6020	0.005	1.0	20	100 ± 25

Table A-1. Estimated Reporting Detection Limits and Precision and Accuracy Objectives for Inorganic Constituents (4 Pages)

Constituent	Analytical Method	Estimated Reporting Detection Limit		Precision (RPD)[b]	Accuracy (% Rec)[c]
		Aqueous (mg/L)	Solids[a] (mg/kg)		
Total Dissolved Solids	160.1	40	N/A	10	100 ± 20[d]
Total Kjedhal Nitrogen	351.2	0.20	N/A	20	100 ± 25
Total Organic Carbon	351.2	0.20	N/A	25	N/A
Total Organic Halides	9020	0.040	0.40	20	100 ± 25
Total Phenolics	420.1	0.15	1.5	20	100 ± 25
Total Phosphorus	365.2	0.030	N/A	20	100 ± 25
Total Residual Chlorine	330.5	0.10	N/A	20	100 ± 25
Total Suspended Solids	160.2	12	N/A	10	100 ± 20[d]
Unsymetrical Dimethylhydrazine	LAL-92-SOP-0189	0.0010	0.0010	15	100 ± 25
Uranium	200.8/6020	0.001	0.20	20	100 ± 25
Vanadium	200.7/6010/CLP	0.05	10	20	100 ± 25
Vanadium	200.8/6020	0.005	1.0	20	100 ± 25
Zinc	200.7/6010/CLP	0.020	4.0	20	100 ± 25

[a] Typically based on 1:10 soil to water ratio
[b] RPD-Relative Percent Difference on the basis of sample and duplicate analyses
[c] Percent recovery of matrix spike sample
[d] Accuracy estimate on the basis of LCS recovery

Table A-2. Reporting Detection Limits for Volatile Organic Analyses by GC/MS (Capillary Column) Using Methods 624/8240A/8260 (2 Pages)

Constituent	Reporting Detection Limit	
	Aqueous (µg/L)	Solid (µg/kg)
Chloromethane	5	5
Vinyl Chloride	5	5
Bromomethane	5	5
Chloroethane	5	5
Trichlorofluoromethane	5	5
Acetone	10	10
2-Chloroethyl vinylether	20	20
1,1-Dichloroethene	5	5
Methylene Chloride	5	5
Carbon Disulfide	5	5
Vinyl Acetate	10	10
1,1-Dichloroethane	5	5
2-Butanone	10	10
trans-1,2-Dichloroethene	5	5
cis-1,2-Dichloroethene	5	5
Chloroform	5	5
1,1,1-Trichloroethane	5	5
Carbon Tetrachloride	5	5
1,2-Dichloroethane	5	5
Benzene	5	5
Trichloroethene (TCE)	5	5
1,2-Dichloropropane	5	5
Bromodichloromethane	5	5
4-Methyl-2-pentanone	10	10
2-Hexanone	10	10
cis-1,3-Dichloropropene	5	5
trans-1,3-Dichloropropene	5	5
1,1,2-Trichloroethane	5	5
Toluene	5	5
Dibromochloromethane	5	5
Tetrachloroethene (PCE)	5	5
Chlorobenzene	5	5
Ethylbenzene	5	5
m,p-Xylene	5	5
o-Xylene	5	5
Styrene	5	5

Table A-2. Reporting Detection Limits for Volatile Organic Analyses by GC/MS (Capillary Column) Using Methods 624/8240A/8260 (2 Pages)

Constituent	Reporting Detection Limit	
	Aqueous (μg/L)	Solid (μg/kg)
Bromoform	5	5
1,1,2,2-Tetrachloroethane	5	5
1,3-Dichlorobenzene	5	5
1,4-Dichlorobenzene	5	5
1,2-Dichlorobenzene	5	5

Table A-3. QC Acceptance Criteria for Matrix Spike and LCS Recoveries for Volatile Organic Analyses by GC/MS using Methods 624\8240A\8260

Matrix Spike Compound	QC Limits (%)			
	Aqueous Sample		Solid Sample	
	% Recovery[1]	RPD[2]	% Recovery[2]	RPD[2]
1,1-Dichloroethene	64-124	14	59-172	22
Benzene	67-127	11	66-142	21
Trichloroethene (TCE)	60-120	14	62-137	24
Toluene	72-132	13	59-139	21
Clorobenzene	68-128	13	60-133	21

[1] - Criteria adopted from Table 7 of SW-846 Method 8260, Revision 0, July 1992.
[2] - Criteria adopted from CLP SOW 3/90 OLM01.0-OLM01.8.

Table A-4. Reporting Detection Limits for Pesticides/PCBs Analyses by GC/ECD Using Methods 608/8080

Constituent	Reporting Detection Limit	
	Aqueous (µg/L)	Solid (µg/kg)
A-BHC	0.05	1.7
B-BHC	0.05	1.7
G-BHC (Lindane)	0.05	1.7
D-BHC	0.05	1.7
Heptachlor	0.05	1.7
Aldrin	0.05	1.7
Heptachlor epoxide	0.05	1.7
G-Chlordane	0.05	1.7
Endosulfan I	0.05	1.7
A-Chlordane	0.05	1.7
4,4'-DDE	0.1	3.3
4,4'-DDT	0.1	3.3
Dieldrin	0.1	3.3
Endrin	0.1	3.3
Endosulfan II	0.1	3.3
4,4'-DDD	0.1	3.3
Endrin Aldehyde	0.1	3.3
Endrin Ketone	0.1	3.3
Endosulfan Sulfate	0.1	3.3
Methoxychlor	0.5	17
Toxaphene	5	170
PCB-1016	1	13
PCB-1221	2	13
PCB-1232	1	13
PCB-1242	1	13
PCB-1248	1	13
PCB-1254	1	13
PCB-1260	1	13
(Technical) Chlordane	1	40

Table A-5. QC Acceptance Criteria for Matrix Spike and LCS Recoveries for Pesticides/PCBs Analyses by GC/ECD Using Methods 608/8080

Matrix Spike Compound	QC Limits (%)			
	Aqueous Sample		Solid Sample	
	% Recovery[1]	RPD[2]	% Recovery[2]	RPD[2]
G-BHC (Lindane)	32-127	15	46-127	50
Heptachlor	34-111	20	35-130	31
Aldrin	42-122	22	34-132	43
Dieldrin	36-146	18	31-134	38
Endrin	30-147	21	42-139	45
4,4'-DDT	25-160	27	23-134	50

[1] Criteria specified in Table 3 of EPA SW-846 for Method 8080, Revision 0, September 1986 which is adopted from Method 608 in 40 CFR Part 136.
[2] Criteria adopted from CLP SOW 3/90, OL01.0-OLM01.8.

Table A-6. QC Acceptance Criteria for Matrix Spike and LCS Recoveries for PCBs Analyses by GC/ECD Using Methods 608/8080

Matrix Spike Compound	QC Limits (%)			
	Aqueous Sample		Solid Sample	
	% Recovery[1]	RPD[2]	% Recovery[1]	RPD[2]
PCB-1260	8-127	30	8-127	50

[1] Criteria specified in Table 3 of EPA SW-846 for Method 8080, Revision 0, September 1986 which is adopted from Method 608 in 40 CFR Part 136.
[2] LAS' best estimates; no criteria specified by Method 608/8080 or CLP SOW.

Table A-7. Reporting Detection Limits for SemivolatileOrganic Analyses by GC/MS Using Methods 625/8270A (2 Pages)

Constituent	Reporting Detection Limit	
	Aqueous (µg/L)	Solids (µg/kg)
Phenol	10	660
bis(2-Chloroethyl)ether	10	660
2-Chlorophenol	10	660
1,3-Dichlorobenzene	10	660
1,4-Dichlorobenzene	10	660
Benzyl alcohol	20	1300
1,2-Dichlorobenzene	10	660
2-Methylphenol	10	660
bis(2-Chloroisopropyl)ether	10	660
4-Methylphenol	10	660
N-Nitroso-di-n-propylamine	10	660
Hexachloroethane	10	660
Nitrobenzene	10	660
Isophorone	10	660
2-Nitrophenol	10	660
2,4-Dimethylphenol	10	660
Benzoic acid	50	3300
bis(2-Chlorethoxy)methane	10	660
2,4-Dichlorophenol	10	660
1,2,4-Trichlorobenzene	10	660
Naphthalene	10	660
4-Chloroaniline	20	1300
Hexachlorobutadiene	10	660
4-Chloro-3-methylphenol	20	1300
2-Methylnaphthalene	10	660
Hexachlorocyclopentadiene	10	660
2,4,6-Trichlorophenol	10	660
2,4,5-Trichlorophenol	10	660
2-Chloronaphthalene	10	660
2-Nitroaniline	50	3300
Dimethylphthalate	10	660
Acenaphthylene	10	660
2,6-Dinitrotoluene	10	660
3-Nitroaniline	50	3300
Acenaphthene	10	660
2,4-Dinitrophenol	50	3300

Table A-7. Reporting Detection Limits for SemivolatileOrganic Analyses by GC/MS Using Methods 625/8270A (2 Pages)

Constituent	Reporting Detection Limit	
	Aqueous (μg/L)	Solids (μg/kg)
4-Nitrophenol	50	3300
Dibenzofuran	10	660
2,4-Dinitrotoluene	10	660
Diethylphthalate	10	660
4-Chlorophenyl-phenylether	10	660
Fluorene	10	660
4-Nitroaniline	20	3300
4,6-Dinitro-2-methylphenol	50	3300
N-Nitrosodiphenylamine	10	660
4-Bromophenyl-phenylether	10	660
Hexachlorobenzene	10	660
Pentachlorophenol	50	3300
Phenanthrene	10	660
Anthracene	10	660
Di-n-butylphthalate	10	660
Fluoranthene	10	660
Pyrene	10	660
Butylbenzylphthalate	10	660
3,3'-Dichlorobenzidine	20	1300
Benzo(a)anthracene	10	660
Chrysene	10	660
bis(2-Ethylhexyl)phthalate	10	660
Di-n-octylphthalate	10	660
Benzo(b)fluoranthene	10	660
Benzo(k)fluoranthene	10	660
Benzo(a)pyrene	10	660
Indeno(1,2,3-cd)pyrene	10	660
Dibenz(a,h)anthracene	10	660
Benzo(g,h,i)perylene	10	660

Table A-8. QC Acceptance Criteria for Matrix Spike and LCS Recoveries for Semivolatile Organic Analyses by GC/MS Using Methods 625/8270A

Matrix Spike Compound	QC Limits (%)			
	Aqueous Sample		Solid Sample	
	% Recovery[1]	RPD[2]	% Recovery[2]	RPD[2]
Phenol	5-112	42	26-90	35
2-Clorophenol	23-134	40	25-102	50
1,4-Dichlorobenzene	20-124	28	28-104	27
N-Nitroso-di-n-propylamine	D-230	38	41-126	38
1,2,4-Trichlorobenzene	44-142	28	38-107	23
4-Chloro-3-methylphenol	22-147	42	26-103	33
Acenaphthene	47-145	31	31-137	19
4-Nitrophenol	D-132	50	11-114	50
2,4-Dinitroltoluene	39-139	38	28-89	47
Pentachlorophenol	14-176	50	17-109	47
Pyrene	52-115	31	35-142	36

[1] Criteria specified in Table 6 of EPA SW-846 for Method 8270A, Revision 1, July 1992 which is adopted from Method 625 in 40 CFR Part 136.

[2] Criteria adopted from CLP SOW 3/90, OLM01.0-OLM01.8.

Table A-9. Reporting Detection Limits for Total Petroleum Hydrocarbons Extractables Analyses by GC/FID Using Method 8015-Modified

Constituent	Reporting Detection Limit	
	Aqueous (mg/L)	Solid (mg/kg)
Diesel	1.0	30
Gasoline	1.0	30

Table A-10. QC Acceptance Criteria for Matrix Spike and LCS Recoveries for Total Petroleum Hydrocarbons Analyses by GC/FID Using Method 8015-Modified

Matrix Spike Compound	QC Limits (%)			
	Aqueous Sample		Solid Sample	
	% Recovery[1]	RPD[2]	% Recovery[1]	RPD[2]
Gasoline	25-145	20	30-130	30
Diesel	25-145[3]	20[3]	30-130[3]	30[3]

[1] LAS-established criteria
[2] LAS' best estimates
[3] Criteria adopted from gasoline analysis

Table A-11. Minimum Detectable Activities for Radionuclide Determinations

Constituent	Minimum Detectable Activity	
	Aqueous (pCi/L)[1]	Solids (pCi/g)
Tritium (H-3)	300	300
Gross α/β[1]	2/4	5/10
Gamma Spec - Nuclide specific (see below)		
Strontium, Isotopic (Sr-89 & Sr-90)	1	1
Strontium, Total	1	1
Uranium, Isotopic	0.5	0.5
Uranium, Total (μg/L or μg/g)	0.1	0.1
Radium-226	0.5	0.5
Radium-228	2.0	N/A
Radium, Total Alpha	2.0	N/A
Radon-222	50	N/A
Technetium-99	1.0	1.0
Thorium, Isotopic	0.5	0.5
Plutonium, Isotopic	0.1	0.1
Americium, Isotopic	0.1	0.1
Cerium, Isotopic	0.1	0.1
Polonium-210	1.0	1
Lead-210	1.0	2
Carbon-14	10	2
Nickel-63	2	2
Iron-55	5	5
Plutonium-241	5	5

[1] For clean water samples (i.e., total solids < 30 mg/250 mL) and if the counting statistics allow.

NOTE: As required, lower MDAs/RDLs can be achieved for all radionuclides.

Table A-12. QC Acceptance Criteria for LCS Recoveries and Duplicates for Radionuclide Determinations

Constituent	LCS Recovery (%)		Relative Percent Difference[1]	
	Aqueous	Solid	Aqueous	Solid[2]
Tritium (H-3)	100 ± 20%	100 ± 20%	20	20
Gross α/β[1]	100 ± 30%	100 ± 30%	30	30
Gamma Spec	100 ± 20%	100 ± 20%	20	20
Strontium, Isotopic (Sr-89 & Sr-90)	100 ± 25%	100 ± 25%	25	25
Strontium, Total	100 ± 25%	100 ± 25%	25	25
Uranium, Isotopic	100 ± 20%	100 ± 20%	20	20
Uranium, Total	100 ± 10%	100 ± 10%	20	20
Radium-226	100 ± 20%	100 ± 20%	20	20
Radium-228	100 ± 30%	N/A	30	N/A
Radium, Total Alpha	N/A	100 ± 20%	N/A	20
Radon-222	100 ± 30%	N/A	30	N/A
Technetium-99	100 ± 25%	100 ± 25%	20	20
Thorium, Isotopic	100 ± 20%	100 ± 20%	20	20
Plutonium, Isotopic	100 ± 20%	100 ± 20%	20	20
Americium, Isotopic	100 ± 20%	100 ± 20%	20	20
Cerium, Isotopic	100 ± 20%	100 ± 20%	20	20
Polonium-210	100 ± 20%	100 ± 20%	20	20
Lead-210	100 ± 20%	100 ± 20%	20	20
Carbon-14	100 ± 25%	100 ± 30%	25	25
Nickel-63	100 ± 20%	100 ± 25%	20	20
Iron-55	100 ± 20%	100 ± 25%	20	20
Plutonium-241	100 ± 20%	100 ± 20%	20	20

N/A: Not applicable

[1] RER ≤ 1, if activity of the sample is less than 10 X MDA.

[2] Solids may have outliers due to potential sample inhomogeneity.

APPENDIX B

GUIDELINES AND REQUIREMENTS FOR SAMPLE CONTAINERS, PRESERVATION, STORAGE, VOLUME, AND HOLDING TIMES

Table B-1. Requirements Containers, Preservation Techniques, Sample Volumes, and Holding Times for Inorganic and Organic Analyses (3 Pages)

Constituent	Analytical Method	Container[1]	Storage & Preservation [2,3]	Minimum Sample Quantity	Maximum Holding Time
INORGANICS:					
Acidity	305.1	P,G	4°C	100 mL	14 days
Alkalinity	310.1	P,G	4°C	100 mL	14 days
Ammonia-N	350.1	P,G	4°C H_2SO_4 pH<2	400 mL	28 days
Bromide	300.0	P,G	None Required	50 mL	28 days
Chemical Oxygen Demand (COD)	410.4	P,G	4°C H_2SO_4 pH<2	50 mL	28 days
Chloride	300.0/325.3/9251	P,G	None Required	50 mL	28 days
Cyanide, total and amenable	335.1/335.4/9010	P,G,T	4°C NaOH pH>12[2] $C_6H_8O_6$	500 mL or 4 ounces	14 days (water & soil)
Filterable Residue (TDS)	160.1	P,G	4°C	100 mL	7 days
Fluoride	340.2	P,G	None Required	300 mL	28 days
Non-Filterable Residue (TSS)	160.2	P,G	4°C	100 mL	7 days
pH	150.1	P,G	None Required	25 mL	Immediate
Total Kjeldahl Nitrogen	351.2	P,G	4°C H_2SO_4 pH<2	500 mL	28 days
Nitrate, as N	300.0/353.2/9200	P,G	None Required	100 mL	48 Hours
Nitrate-Nitrite	300.0/353.3	P,G	4°C H_2SO_4 pH<2	100 mL	28 days
Nitrite, as N	300.0/351.2	P,G	None Required	100 mL	48 hours
Orthophosphate, as P	365.2	P,G	Filter; 4°C	50 mL	48 hours
Total Phosphorous, as P	365.2	P,G	4°C H_2SO_4 pH<2	50 mL	28 days
Specific Conductance	120.1/9050	P,G	None Required	100 mL	28 days
Temperature	170.1	P,G	None Required	1000 mL	Immediate
Total Hardnes ($CaCO_3$)	200.7	P,G	4°C HNO_3 pH<2	100 mL	180 days
Total Organic Carbon	415.1/9060	P,G,T	4 °C HCl/H_2SO_4 pH<2[2]	500 mL or 4 ounces	28 days (water and soil)
Turbidity	180.1	P,G	4°C	100 mL	48 hours
METALS: Chromium+6	7196	P,G,T	4°C	500 mL or 8 ounces	24 hours[4] (water and soil)
Mercury	245.2/7470/7471	P,G,T	4°C HNO_3 pH<2[2]	500 mL or 8 ounces	38 days in glass 13 days in plastic 28 days for CLP/TCLP

Table B-1. Requirements Containers, Preservation Techniques, Sample Volumes, and Holding Times for Inorganic and Organic Analyses (3 Pages)

Constituent	Analytical Method	Container[1]	Storage & Preservation [2,3]	Minimum Sample Quantity	Maximum Holding Time
INORGANICS:					
All metals (except Cr^{+6} and Hg)	200.7/6010; 200/7000 Series	P,G,T	4°C HNO_3, to pH < 2[2],	500 mL or 8 ounces	180 days (water and soil)
ORGANICS:					
Total Recoverable Petroleum Hydrocarbons (TRPH)	418.1	G,T	4°C H_2SO_4 pH < 2	1000 mL or 8 ounces	28 days (water and soil)
Oil and Grease	413.1/413.2/ 9070/9071	G	4°C H_2SO_4 pH < 2	1000 mL or 8 ounces	28 days (water and soil)
Total Petroleum Hydrocarbons-*Gasoline*	8015 (Modified)	G	4°C $Na_2S_2O_3$; HCl pH < 2	1000 mL or 8 ounces	14 days (water and soil)
Total Petroleum Hydrocarbons-*Diesel*	8015 (Modified)	G	None required	1000 mL or 8 ounces	7 days to extraction; 40 days to analysis
Purgeable Halocarbons	8010	G, Teflon-lined septum	4°C $Na_2S_2O_3$	3x40 mL	14 days
Aromatic Volatile Organics	8020	G, Teflon-lined septum	4°C HCl pH < 2[2]; $Na_2S_2O_3$	3x40 mL or 4 ounces	14 days (water and soil)
Chlorinated Herbicides	8150	G, Teflon-lined septum	4°C pH 5-9	1000 mL or 8 ounces	Water - 7 days to extraction; 40 days to analysis Soil - 14 days to extraction; 40 days to analysis
Pesticides and Polychlorinated Biphenyls (PCBs)	8080/8140	G, Teflon-lined cap	4°C pH 5-9	1000 mL or 8 ounces	Water - 7 days to extraction; 40 days to analysis Soil - 14 days to extraction; 40 days to analysis
Phenols	8040	G, Teflon - Lined, cap	4°C $Na_2S_2O_3$	1000 mL or 8 ounces	Water - 7 days to extraction; 40 days to analysis Soil - 14 days to extraction; 40 days to analysis
Semivolatile Organics	625/8270	G, Teflon-lined cap	4°C $Na_2S_2O_3$	1000 mL or 8 ounces	Water - 7 days until extraction; 40 days to analysis Soil - 14 days until extraction; 40 days to analysis
Volatile Organics	8240/8260	G, Teflon - lined septum	4°C $Na_2S_2O_3$ HCl pH < 2[2]	3 x 40 mL or 4 ounces	14 days (water and soil); 7 days (water-unpreserv

Table B-1. Requirements Containers, Preservation Techniques, Sample Volumes, and Holding Times for Inorganic and Organic Analyses (3 Pages)

Constituent	Analytical Method	Container[1]	Storage & Preservation [2,3]	Minimum Sample Quantity	Maximum Holding Time
INORGANICS:					
Polycyclic Aromatic Hydrocarbons (PAHs)	8310	G, Teflon - lined cap	4°C $Na_2S_2O_3$	1000 mL or 8 ounces	Water - 7 days until extraction; 40 days after extraction Soil - 14 days until extraction; 40 days after extraction
Toxicity Characteristic Leaching Procedure (TCLP)	1311	G, teflon - lined cap	Cool, 4°C	1000 mL or 8 ounces	VOAs - 14 days to TCLP extraction SemiVOAs - 14 days to TCLP extraction and 40 days to analysis Mercury - 28 days to TCLP extraction; 28 days to analysis Metals - 180 days to TCLP extraction; 180 days to analysis
Explosives	8330	P, G, T	Cool, 4°C	1000 mL or 8 ounces	Water - 7 days to extraction Soils - 14 days to extraction Analyze w/in 40 days

1 Polythylene (P); Glass (G); Brass sleeves.

2 No pH adjustment is required for soils.

3 Preservation with 0.008 % $Na_2S_2O_3$ is only required when residual chlorine is present.

4 Holding time requirement for Chromium

6 in soils has not been established. The recommended hold. time for extracting into water is 48 hours. The sample must be analyzed w/in 24 hrs of extraction.

Sources: Table 2-21 in EPA SW-846 (Revision 1, July 1992); EPA-600/4-79-020 (Revised March 1983); CLP SOWs.

APPENDIX C

STANDARD OPERATING PROCEDURES INDEX

SOP INDEX

Number	Document Title
LAL-90-SOP-0001	Document Control
LAL-90-SOP-0002	Sample Receipt and Login
LAL-90-SOP-0003	Waste Handling and Disposal
LAL-90-SOP-0004	Preventing Sample Contamination
LAL-90-SOP-0005	Standards Traceability in the Inorganic and Organic Laboratories
LAL-90-SOP-0006	Maintenance of Laboratory Logbooks and Records
LAL-90-SOP-0007	[illegible]
LAL-90-SOP-0008	Technical and Managerial Review of Analytical Data
LAL-90-SOP-0009	Internal Sample Chain-of-Custody and Evidentiary Procedures
LAL-90-SOP-0010	Internal System Evaluation
LAL-90-SOP-0011	[illegible]
LAL-90-SOP-0012	Independent QA Validation of Inorganic Analysis Data
LAL-90-SOP-0013	Independent QA Validation of Organic Analysis Data
LAL-90-SOP-0014	Security
LAL-90-SOP-0015	Routine and Periodic Maintenance of Laboratory Support Equipment
LAL-90-SOP-0016	Analysis of VOCs by GC-MS (EPA 624/8240/8260/CLP)
LAL-90-SOP-0017	Glassware Cleaning in the Organic Chemistry Laboratory
LAL-90-SOP-0018	Glassware Cleaning for Inorganic Analyses
LAL-90-SOP-0019	Procedure for the Determination of Anions by Ion Chromatography
LAL-90-SOP-0020	Determining Ammonia in Surface Water and Waste Water by Flow Injection Analysis
LAL-90-SOP-0021	Determination of Specific Conductance
LAL-90-SOP-0022	Determination of the pH
LAL-90-SOP-0023	Analysis of Volatile Organic Compounds in Drinking Water by Capillary Column GC/MS
LAL-90-SOP-0024	Analysis of Semivolatile Organics Compounds by Capillary Column GC/MS
LAL-90-SOP-0025	Analysis of Purgeable Aromatics and Halocarbons in Wastewater and Drinking Water by Gas Chromatograph Using Methods 8010/8020, 601/602, and 502.2
LAL-90-SOP-0026	Continuous Liquid-Liquid Extraction on Organic Compounds
LAL-90-SOP-0027	Separatory Funnel Extraction of Organic Compounds
LAL-90-SOP-0028	Extractable Organics in Soil/Solid Matrices by Sonication Extraction
LAL-90-SOP-0029	Extractable Organics in Soil/Solid Matrices Soxhlet Extraction
LAL-90-SOP-0030	Analysis of Volatile Organic Compound in Drinking Water by Gas Chromatography (Hall/PID)
LAL-90-SOP-0031	Analysis of Trihalomethanes in Drinking Water by GC/MS Selected Ion Monitoring

SOP INDEX

Number	Document Title
LAL-90-SOP-0032	Block Digestor Sample Preparation for the Determination of Metals by Inductively Coupled Plasma Emission Spectroscopy
LAL-90-SOP-0033	Sample Preparation for the Determination of Metals by Graphite Furnace AAS (GFAAS)
LAL-90-SOP-0034	Procedure for Sample Preparation for the Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy (CVAAS)
LAL-90-SOP-0035	Procedure for Determination of Percent Solid in Soils, Sediments, and Sludges
LAL-91-SOP-0036	Determination of Total Hardness of Water by EDTA Titration
LAL-90-SOP-0037	Determination of Metals by Inductively Coupled Plasma-Atomic Emission Spectrometer (ICP-AES) Using Leeman Model PS-3000 ICP-AES
LAL-90-SOP-0038	Determination of Metals by GFAAS
LAL-91-SOP-0039	Determination of Sulfide in Water
LAL-90-SOP-0040	Determination of Total Filterable Residue
LAL-91-SOP-0041	Data Completeness and Client Compliance Review
LAL-90-SOP-0042	Potentiometric Determination of Fluoride in Aqueous Samples
LAL-90-SOP-0043	[illegible]
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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

QUALITY ASSURANCE MANUAL

for

SOUTHWEST LABORATORY
OF OKLAHOMA, INC.
Broken Arrow, Oklahoma

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APPROVAL:

QA/QC Officer Chuck Hoover Date : April 28, 1995

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1.0 QUALITY ASSURANCE

1.1 INTRODUCTION

This document details the quality assurance plan employed by Southwest Laboratory of Oklahoma (SWLO) to address various client and contract requirements necessary to provide service as an analytical laboratory. It details laboratory procedures with emphasis on the Quality Assurance/ Quality Control (QA/QC) requirements based on EPA guidelines for the analysis of multimedia samples for a broad range of inorganic and organic contaminants. It reflects the necessary specifications to assure accuracy, precision, completeness, representativeness, and comparability on all tasks of the contract or project. The purpose of this plan is to assure that data of the highest quality are being reported by SWLO.

1.2 DEFINITION OF TERMS

Quality Assurance

A quality assurance program is an essential part of a sound analytical protocol used by individuals and laboratories to detect and correct problems in the measurement process or to demonstrate attainment of a state of statistical control. The objective of a quality assurance program in analysis is to reduce measurement errors to agreed-upon limits and to produce results of acceptable quality. Two concepts are involved in quality assurance: (1) quality control, the mechanism established to control errors, (2) quality assessment, the system used to verify that the analysis is operating within acceptable limits.

Quality Control

A quality control program includes the following:

- Development of and strict adherence to principles of good laboratory practice,
- Consistent use of standard operation procedures,
- Establishment of and adherence to carefully designed protocols for specific measurement programs,
- The consistent use of qualified personnel,
- Reliable and well-maintained equipment,
- Appropriate calibrations and standards,
- The close supervision of all operations by management and senior personnel.

When properly conceived and executed, a quality control program will result in a measurement system operating in a state of statistical control, which means errors have been reduced to acceptable levels and have been characterized statistically.

Quality Assessment

Quality assessment includes a variety of techniques required to assess the quality of the measurement process and the results. The establishment of a system of "control charts" is a basic principle. Control charts are plots of multiple data points from the same or similar samples or processes versus time. They are used to determine if a system is in a state of statistical control. Control charts should be used to visualize or monitor the relative variability of repetitive data. They can be used with reference materials, spiked samples, and the analysis of surrogates as a means to assessing the accuracy of measurements.

Quality Assessment Procedures

Procedures used to assess the effectiveness of the quality control system include:

(a) Internal Performance Audits—conducted by the use of control samples, replicate measurements and use of reference materials in conjunction with control charts

(b) External Performance Audits—conducted by the use of inter-laboratory checks such as:

- Participation in laboratory evaluation programs; State Programs (Utah, Florida, California, Oklahoma, etc.), and Corps of Engineers D.E.R.A. Program.

- Participation in performance evaluation samples available from EPA (WP & WS Studies).

Quality Assessment Procedure Summary

A simplified working document or chart which enables one to preview the basic quality control program and its effectiveness shall be maintained on a daily basis. The following parameters are to be tracked:

- Areas of the internal standard for all samples,

- Surrogate spike recovery for all samples,

- Matrix spike and matrix spike duplicate sample results for accuracy and precision.

QA Organization

Assembled data shall be reviewed by the Project Officer before technical compilation into contract deliverables. Final review of about 20% of the assembled deliverables package is performed by the Quality Assurance Officer (QA Officer).

Data Quality

Data quality is the totality of features and characteristics of data that bear on their ability to satisfy a given purpose. Parameters of major importance are accuracy, precision, completeness, representativeness, and comparability. These are defined as follows:

(a) Accuracy - The degree of the difference between measured or calculated values and true value.

(b) Precision - The reproducibility or degree of agreement among replicate measurements of the same quantity.

(c) Completeness - The percentage of valid data obtained from a measurement system.

(d) Representativeness - The degree to which the data accurately represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

(e) Comparability - The confidence with which one data set can be compared to another.

1.3 ORGANIZATION AND RESPONSIBILITIES

Corporate Organization

SWL is located at 1700 West Albany, Broken Arrow, Oklahoma 74012, with additional facilities located in Tulsa, Oklahoma, Cushing Oklahoma, Baton Rouge, Louisiana, and St. Louis, Missouri.

Specific QA/QC responsibilities are summarized in the following subsections. Figure 1.1 shows the line-staff relationships within the laboratory.

Laboratory Director

The Laboratory Director has overall responsibility for the technical quality, cost control, laboratory personnel management, and adherence to project schedules. His overall management involves the quality assurance of the following items:

- Delivery order/work assignments

- Adherence to delivery schedules

- Deliverable reports
- Subcontractor work product if required

- Project/contract cost control and accounting

- Task performance of key personnel

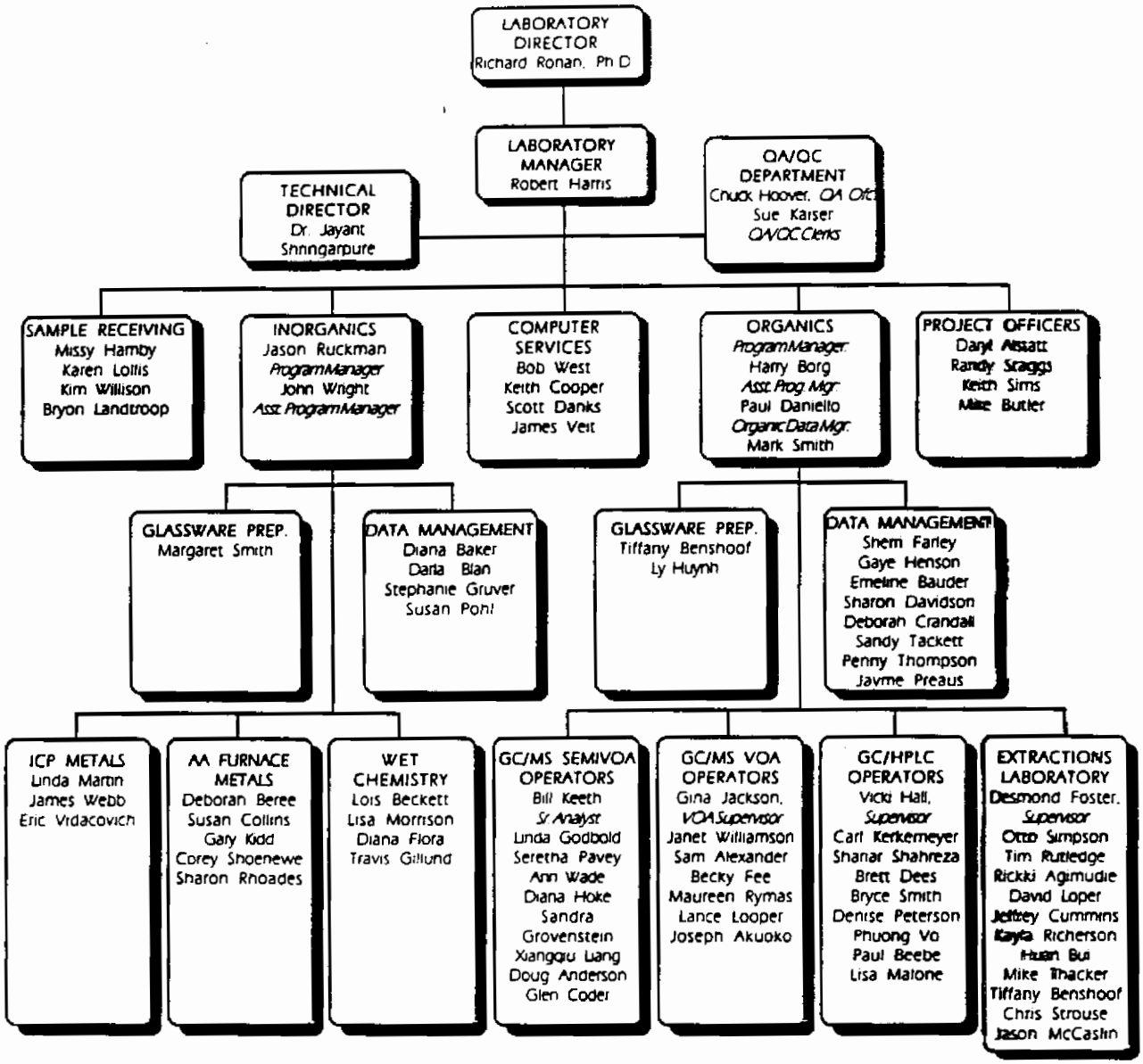

FIGURE 1.1
Laboratory Organizational Chart

Laboratory Manager

The Laboratory Manager assists the Laboratory Director as the need arises, and is closely involved with the day-to-day activities of sample preparation/analyses. The Laboratory Manager coordinates all laboratory activities necessary to fulfill contract requirements.

Quality Assurance Officer

The QA Officer is responsible for monitoring the quality of laboratory work and taking appropriate actions to ensure that quality standards are being met. The QA Officer reports directly to the Laboratory Manager in reviewing the work of teams and individuals. The QA Officer is responsible for the following:

■ Preparing and overseeing the preparation of the laboratory QA/QC plan;

■ Establishing QC procedures and setting warning and action limits for every test or parameter to standardize laboratory operation for quality performance;

■ Coordinating State & Federal Performance Evaluation Studies.

■ Monitoring compliance with the laboratory's QA/QC plan by:

1. Reviewing QC-related activities and documentation for completeness in accordance with the QA/QC plan

2. Identifying and referring any instances in which the QA/QC objectives are not being met to Group Leaders or Laboratory Supervisors for remedial and/or corrective action.

Analytical Program Manager

The Analytical Program Manager oversees the primary functions of this group: sample control, document control, data management, and client services. The Program Manager provides supervision and guidelines for sample handling and storage prior to analyses, maintenance of project files, data entries into the computer system after sample analysis, and quality review of the final data deliverables. Under the Program Manager, the following personnel play a vital role in the QA/QC program:

■ Project Officer (PO) - responsibilities include direct client representation within the laboratory. The PO acts in an advocacy role for the client. As needed, the PO will review with clients the technical aspects of the analytical results and how they relate to the needs of the client. The PO will monitor all analytical projects as they progress through the laboratory.

■ Sample Custodian (SC) - handles all sample receipt, adheres to chain-of-custody procedures, does computerized sample log-in, assigns secure sample storage for samples and initiates the set-up of the project file. The SC is responsible for maintaining the integrity and validity of the samples.

■ Data Clerk - responsibilities include: document organization, assembly of all documents relating to contracts or projects (including project file and analytical file) to ensure clerical veracity during data-handling, data assembly, and data report production.

Inorganic and Organic Supervisors

These Laboratory Supervisors have primary responsibility for the technical quality of all data generated within their respective sections. In addition they are responsible for the adherence to delivery schedules, management and utilization of manpower and the technical aspects of all Standard Operating Procedures (SOPs).

Systems Manager

Responsible for the management and quality control of all computing systems (hardware, software, documentation and procedures), generating, updating, and performing quality control reviews of automated deliverables.

Programmer Analyst

Responsible for the installation, operation, and maintenance of software and programs, generating, updating and performing quality control reviews of analytical databases and automated deliverables.

1.4 CERTIFICATIONS AND ACCREDITATIONS

The SWLO laboratory has participated in available certification programs pertaining to environmental chemistry. Table 1.2 lists the various certifications and accreditation programs in which SWLO participates.

1.5 EQUIPMENT AND SUPPLIES

Procurement and Inventory

To assure a good laboratory quality assurance program in the procurement of equipment and supplies, SWLO has established criteria and specifications for the purchase of important pieces of equipment. Factors such as cost, volume of work, ease of operation, inherent accuracy, expected equipment lifetime, length and condition of warranties or service contracts, expected downtime and repair costs are considered in the basis of selection. The increased usage of electronic analytical instruments has improved the quality and quantity of data and has increased productivity.

Control of materials (i.e., reagents, standards, solvents, etc.) and glassware used in the analyses is maintained as part of the quality assurance program. Reagents & Solvents are analyzed prior to use to verify purity; documentation of these analyses are maintained. Lot numbers are recorded on extraction logs to facilitate the tracking of these items. All reagents are dated as they are received and when they are opened to ensure systematic use. The identity, purity, shelf-life, source, tests to be conducted for quality and purity, storage and handling procedures, and replacement dates are factors that are considered in making purchase requisitions.

Before any purchases are made, purchase requisition orders and requests are checked and verified by the Laboratory Supervisors. Approval for such purchases is made by the Laboratory Supervisors, and the Laboratory Director.

Equipment Management

Information on performance of the equipment is obtained before a purchase request is made. Service availability for installation specification and verification is considered in purchase negotiation. When the instrument/equipment is installed, an internal calibration is made on the instrument to meet manufacturer's specifications. Calibration checks are done by using analytical reference standards for qualitative and quantitative checks to verify instrument performance during the sample run. Routine preventive maintenance of the instruments or equipment is made on a regular basis. Section 9.2 discusses preventive maintenance employed by the different laboratory sections to ensure instrument/equipment working conditions.

The laboratory maintains sufficient inventory of analysis and/or testing equipment to meet client requirements for analytical services. Table 1.3 contains current lists of instrumentation and associated equipment which the laboratory uses in providing analytical services.

Supplies Management

Materials, reagents, solvents and gases are carefully selected to meet specifications as prescribed by the method of analyses. Each new supply of these items is verified for performance capability based on the required certified assay/analysis of chemicals and freedom from impurities that would interfere with the analysis. Background levels are measured to check the degree of contamination. In storage, these items are protected from degradation and contamination through conformance of storage requirements according to the manufacturer's directions and/or individual method of analysis. Solvents used for extraction are pre-analyzed to determine impurities that might interfere with the analytes of interest. (See Appendix D for Standard Operating Procedures for Checking Analytical Solvents). Standards and reagents are dated upon receipt, and the date of expiration recorded. This procedure establishes the order of use and eliminates the possibility of exceeding shelf-life. (See Appendix D for Standard Operating Procedure for Standard Expiration)

Source of Standard Reagents

Primary standards and/or stock standards are obtained from a reliable, certifiable source and are high purity. Standards are purchased from approved commercial vendors such as Chem Services, Fisher Scientific, Supelco, Altrex, etc., for use in all analytical testing. Standards are protected from degradation, deterioration, and contamination based on storage requirements (e.g. polyethylene containers for alkali solutions, glass

containers for organics and brown glass for light-sensitive solutions; temperature storage and segregation of standards based on reactivity). Prepared commercial standards are verified against certified standard reference materials, (SRM) from EPA or NBS for traceability. (See Appendix D for Standard Operating Procedure for Traceability of Standards)

Stock and working standard solutions are prepared fresh as required by their stability, and are checked regularly for signs of deterioration, (i.e., discoloration, formation of precipitates, and changes in concentration). Standard solutions are labeled with compound name, concentration, solvent, date of preparation, and preparer. (See Appendix D for Standard Operating Procedure for Standard Expiration)

Glassware

Class A volumetric glassware is used by the laboratory for measuring trace constituents in both inorganic and organic analysis.

Laboratory contamination is minimized through implementation of a standard operating procedure (SOP) for glassware and labware cleaning. It is followed to ensure the removal of all traces of parameters of interest and contaminants that could interfere with analysis. (See Appendix D for Standard Operating Procedure for Glassware and Labware Cleaning)

Reagents, Solvents and Gases

Chemical reagents aside from the primary standard reagents, solvents and gases are carefully selected to conform to specifications defined in the method of analyses. Selection is based on the required priority for parameters being measured, sensitivity of the method and specificity of the detection system (i.e., AA, ICAP, GC-ED, GC/MS).

Laboratory reagents obtained from approved commercial vendors shall meet ACS standards and are labeled indicating contents, date of receipt or preparation, and expiration. Hazardous reagents are adequately labeled and stored segregated from the rest of the reagents to indicate type and degree of hazard. (See Appendix D for Standard Operating Procedure for Standard Expiration)

Solvents to be used for extraction are pre-analyzed to detect the presence of impurities which might possibly interfere with analytes of interest. When a particular lot is found to be acceptable, the manufacturer is notified to set aside a specified number of cases of this lot for SWLO laboratory usage. The solvent is checked on a regular basis to ensure high level of solvent quality control is maintained within the lot. (See Appendix D for Standard Operating Procedure for Checking Analytical Solvents). All solvents are dated upon receipt and again upon opening to ensure first-in — first out useage. Solvent bottles are stored in a grounded flameable liquid storage cabinet.

Gases used in inorganic and organic analyses are of commercial grade or are laboratory-supplied gases. For organic analyses, the type of detection (i.e., GC-EC, Hall, GC-FID, GC/MS) used affects gas quality requirement. Molecular sieves carrier-gas filters, and drying tubes are required on combustion gases to improve quality. Gas cylinders are immediately replaced when the pressure falls to 100-200 pounds per square inch (psi) to minimize detector contamination that will affect sensitivity of the detector.

Laboratory Reagent Water

ASTM Type II water is used in the laboratory for dilution, preparation of reagent solutions and final rinsing of glassware. It is free from interferences and other contaminants. After passing through two ion exchange canisters and one carbon filter canister, water purity is monitored by an indicator light at each outlet and at the filtration apparatus. (See Appendix D, Preparation and QA/QC for the Laboratory Water Supply)

Compressed Air

Compressed air is employed mainly in instruments using GC oven-door control and autosamplers. Absorption filters are installed between the outlet and the point of use to trap oil, moisture, and other contaminants entering the compressed air transfer lines. These lines are checked periodically for the presence of moisture and contaminants and are replaced as soon as moisture is detected.

Hood System

An efficient hood system is necessary to remove the various toxic and hazardous fumes that may be generated when using organic solvents or that may be formed during an acid digestion step. It is also used to remove toxic gases that may be formed during atomic absorption analysis reactions. The laboratory fume hood face velocity is regularly checked every four months for optimum face velocity.

Electrical Services

The laboratory electrical system provides adequate and constant voltage, appropriate grounding, and efficient lighting which is required for satisfactory lighting, proper functioning of sensitive instruments and operation of high-current devices. A licensed electrical contractor provides repairs and services in the event of a power failure or electrical problems.

Computer Capabilities

Computer systems in current use include the following:

- Microcomputers - (IBM, AT, XT, Compaq, etc.) are used primarily for data processing. These may also be directly interfaced with instrumentation.

- Minicomputers - Used primarily for instrument control.

- LIMS System - A laboratory information management system. This system is attached to our local area network system and is used for sample log-in, sample tracking, data reports, invoicing, and management reports.

1.6 QUALITY ASSURANCE OBJECTIVES

The purpose of SWLO's quality assurance plan is to ensure that the laboratory provides high-quality and cost-effective services and products to its clients. Although specific quality assurance procedures will be designed to meet the needs of each individual program, SWLO's general objectives are:

- Data should be accurate in terms of their agreement with a reference or true values.

- Data should be precise in that there is agreement among individual measurements made under similar condition

- Data should be complete in terms of the amount of data available vs. the amount of data evaluated.

- Data should be comparable to prior relevant data for evaluation and testing purposes.

- Data should be representative of the overall population or data base of parameter measurements.

- Data should be reproducibly obtainable under similar conditions, whether generated by the SWLO or another firm.

- Upgrade the overall quality of laboratory performance.

All of the above objectives are ensured by the QA/QC program which monitors all phases of data generation, ranging from sample collection to sample handling, to the actual analysis and data reporting that involves measurements of both inorganic and organic constituents. These procedures will be followed by all personnel and will routinely be reviewed by both the Laboratory Director and QA Officer.

Scope and Approach Relating to Measurement of Data in Terms of Precision, Accuracy, Completeness, Representativeness, and Comparability

The laboratory scope and approach to produce data of known and sufficient quality are described in this section. Guidelines are provided for the assessment and reporting of data quality for any environmentally related measurements, and for the incorporation of such assessments into major environmental data bases.

Controlled sample receiving, logging, and tracking throughout the length of the project/contract is maintained to ensure sample integrity throughout the sample analysis scheme. Documentation of instrument performance and preventive maintenance is used to provide a permanent record for data validation. SWLO routinely checks the quality of analytical work through analysis of quality control (QC) reference samples, duplicate samples, or matrix spike duplicate and spike samples.

Accuracy

The accuracy of the measurement data is evaluated by the comparison of the percent recovery of the QC reference material of known or established concentration, independent of routine calibration. Statistically based control limits are established for each method of analysis and sample matrix. A spike sample is analyzed routinely for each batch of 20 samples (5%) and are dependent upon the sample matrix, method of analysis and concentration level. A more frequent analysis is performed (i.e., one in 10 samples) on a contract-specific basis. Recoveries are assessed to determine method efficiency and matrix interference effects. Analytical accuracy is expressed as the percent of recovery of an analyte/parameter which has been added to the environmental samples at a known concentration before preparation and analysis. The equation used to calculate percent recovery is

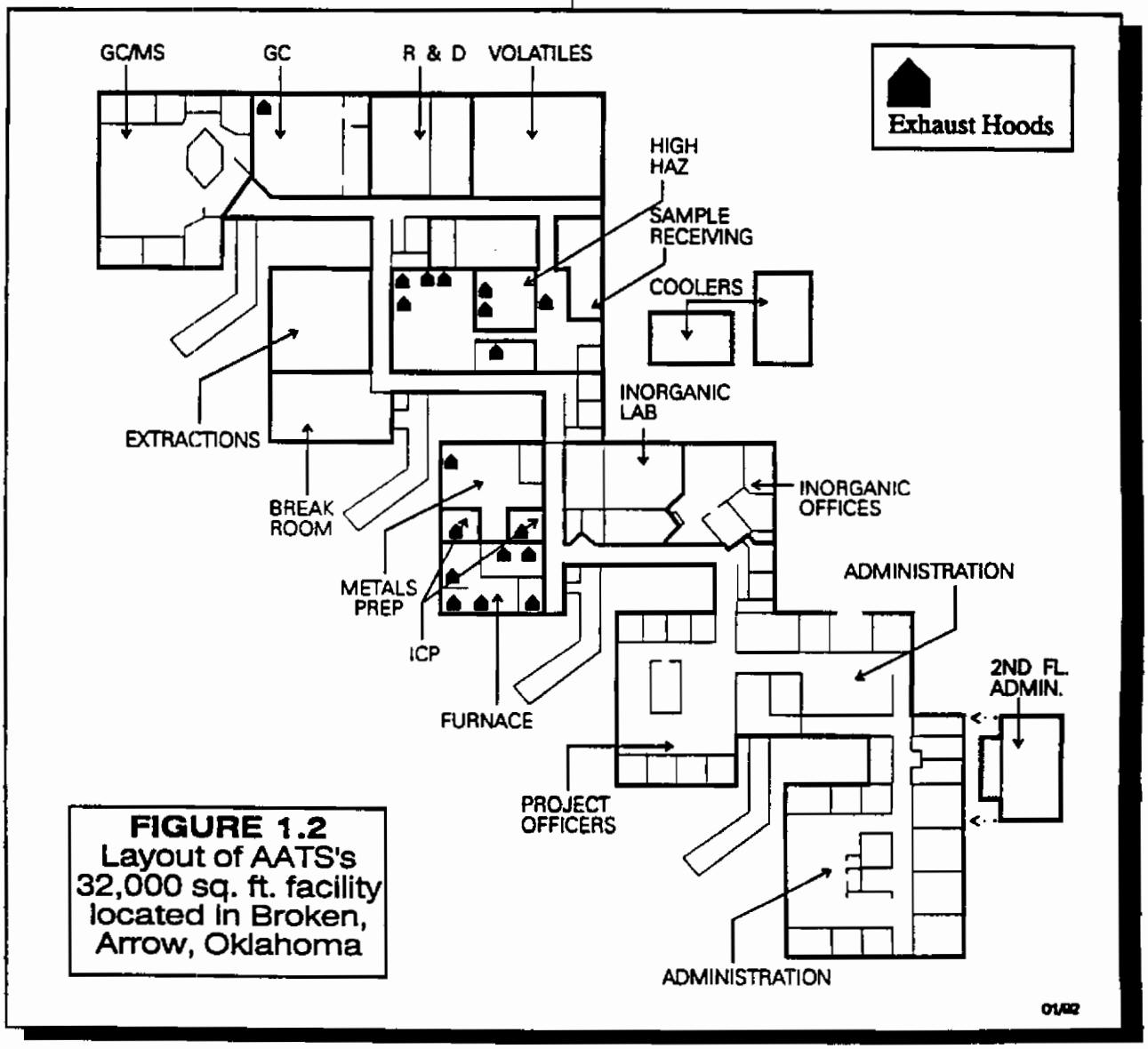

FIGURE 1.2
Layout of AATS's 32,000 sq. ft. facility located in Broken, Arrow, Oklahoma

as follows:

$$\text{Percent Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result}) \times 100}{\text{Amount of Spike Added}}$$

Precision

The laboratory uses matrix spike duplicates or duplicate sample analysis to assess precision. A matrix spike duplicate or a duplicate sample is analyzed for each batch of 20 samples (5%) for in-house QC and is dependent upon the sample matrix and method of analysis. A more frequent analysis is performed (i.e., one in 10 samples) on a contract-specific basis. The basic precision statistics obtained from the multiple batch frequency may be compared to develop a graph assessment (using control limits) for given sample matrix.

Analytical precision is expressed as a percentage of the difference between the results of two matrix spike samples or two duplicate sample analysis for a given analyte. Relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{(\text{MS Result} - \text{MS Duplicate Result}) \times 100}{\text{Mean of MS and MS Duplicate Results}}$$

where MS denotes Matrix Spike.

or

$$RPD = \frac{\left(\text{Sample Result} - \text{Duplicate Result}\right) \times 100}{\text{Mean of Sample and Duplicate Results}}$$

Completeness

For the data to be valid, it must meet all the acceptance criteria including accuracy, precision and any other criteria specified by the analytical method used. Data validation procedures are employed to minimize the amount of bad data from getting through data collection.

While the quality objective is to obtain the greatest accuracy and precision, the specific accuracy/precision level is dependent on the method of analysis and type of sample matrix. The laboratory historical statistical control limits (see Appendix E) are used as guidelines to validate the data generated unless client or contract requirements set more stringent criteria.

Representativeness

Data generated by the laboratory shall be representative of the overall population of samples collected and analyzed. It shall be representative of the laboratory data base of accuracy and precision measurements of the particular parameter(s), matrix and analytical method. If the same results are reproducible, the data can be said to represent the environmental condition.

Comparability

Data generated shall be used to evaluate completeness of extensive monitoring programs and testing purposes based on the previous data measurements of parameters, matrix and analytical method. It shall be comparable to data sets recorded in the past to check for historical consistency. In order to maximize its usefulness, data shall be reported in appropriate units and in a consistent manner. Appropriate units are in accordance with the requirements stated in the October 26, 1984, proposed rules 40 CFR, Part 136, Guidelines Establishing Test Procedures for the Analysis of Pollutants. Data should be reproducible under similar conditions whether generated by the laboratory or another firm.

TABLE 1.0

Technical Staff and Experience

RICHARD J. RONAN
Laboratory Director
Ph.D, Inorganic Chemistry

GC/MS	2 years
ICP	20 years
Gas Chromatography	15 years
Atomic Absorption	20 years
Data Review	20 years
Inorganic Analysis	20 years

ROBERT HARRIS, Laboratory Manager
B.S., Microbiology

GC/MS	4 years
ICP	1 year
Purge and Trap	3 years
Gas Chromatography	10 years
Atomic Absorption	15 years
Sample Preparation	15 years
Data Review	15 years

DR. JAYANT SHRINGARPURE
Technical Director
Ph.D., Organic Chemistry

GC/MS	12 years
Purge & Trap	12 years
Gas Chromatography	15 years
High Perf. Liquid Chromatography	5 years
Sample Preparation	5 years
Data Review	12 years

CHUCK HOOVER, QA/QC Officer
B.A., Biology, (Chemistry Minor)

GC/MS	2 years
Purge and Trap	2 years
Gas Chromatography	5 years
Atomic Absorption	4 years
Sample Preparation	8 years
Data Review	6 years

HARRY BORG, Organic Program Mgr.
B.A., Chemistry/Minor: Physics, Math

GC/MS	7 years
Purge & Trap	1 year
Gas Chromatography	10 years
Atomic Absorption	1-1/2 years
Sample Preparation	2 years
Data Review	7 years
Inorganic Analysis	2-1/2 years
High Perf. Liq. Chromatography	1-1/2 years

PAUL DANIELLO, Ass. Organic Prog. Mgr.
B.A., Biology

GC/MS	9 years
Purge & Trap	11 years
Gas Chromatography	11 years
Sample Preparation	11 years

MARK SMITH, Organic Data Manager
B.S., Chemistry

GC/MS	5 years
Purge & Trap	5 years
Sample Preparation	1 year
Data Review	3 years
Inorganic Analysis	0.5 years

DARYL ALSTATT, Project Officer
B.A., Chemistry

Sample Preparation	5 years
Data Review	4 years
Inorganic Analysis	5 years

KEITH SIMS, Project Officer
B.S., Biology, (Chemistry Minor)

ICP	4 years
Atomic Absorption	10 years
Sample Preparation	12 years

RANDY STAGGS, Project Officer
B.S., Biology

Gas Chromatography	3 years
Atomic Absorption	5 years
Sample Preparation	5 years
Data Review	10 years
Inorganic Analysis	15 years

8/94

JASON RUCKMAN
Inorganics Program Manager
B.S., Chemistry

ICP	4 years
Atomic Absorption	5 years
Sample Preparation	4 years
Data Review	4 years

JOHN WRIGHT
Asst. Inorganic Prog. Manager
B.S., Chemistry

Atomic Absorption	3 years
Sample Preparation	3 years
Data Review	3 years
Inorganic Analysis	5 year
ICP	2 years

LOIS BECKET, Inorganic Analysis
B.A., Chemistry

Gas Chromatography	2 years
Atomic Absorption	3 years
Sample Preparation	12 years
Inorganic Analysis	16 years
Organic Extractions	5 years

DEBORAH BEREE, Wet Chemistry
B.S., Biochemistry

Atomic Absorption	2 years
Inorganic Analysis	2 years

SUSAN COLLINS, AA Furnace
A.S., Chemistry (pending 5/92)

ICP	1 year
Gas Chromatography	2 years
Atomic Absorption	11 years
Data Review	10 years
Inorganic Analysis	10 years
High Perf. Liq. Chromatography	1 year

DIANA FLORA, Inorganic Analysis
H.S. Diploma

Inorganic Analysis	1 year
Sample Preparation	2 years

GARY KIDD, AA Furnace
H.S. Diploma

Atomic Absorption	2 years
Sample Preparation	2 years
Inorganic Analysis	2 years

LISA MORRISON, Inorganic Analysis
H.S. Diploma

Sample Preparation	3 years
Inorganic Analysis	3 years

COREY SHOENEWE, AA Furnace
B.S., Biomedical Science

Sample Preparation	1 year
Atomic Absorption	1 year

JAMES WEBB, AA Furnace
B.S., Biology

Atomic Absorption	6 years

ERIC VIDACOVICH
ICP Trace Analysis
3 years college, Biology/Chemistry

ICP	5 years
Atomic Absorption	8 years
Sample Preparation	8 years
Data Review	6 years

GINA JACKSON,
GC/MS Volatile Organics Section Supv.
B.S., Chemistry

GC/MS	4 years
Purge & Trap	1 year
Sample Preparation	1 year

SAM ALEXANDER, GC/MS Laboratory
B.S. Chemistry

GC/MS	2 years
Gas Chromatography	4 years
Atomic Absorption	3 years
Sample Preparation	3 years
Data Review	3 years

REBECCA FEE, GC/MS Laboratory
B.S., Chemistry

GC/MS	3 years
Purge & Trap	5 years
Gas Chromatography	3 years
High Perf. Liq. Chorm.	3 years

MAUREEN RYMAS, GC/MS Laboratory
Certified Lab Technician

GC/MS	1 year
ICP	2 years
Atomic Absorption	4 years
Sample Preparation	4 years
Purge & Trap	1 year

JANET WILLIAMSON, GC/MS Laboratory
M.S., Analytical Chemistry

GC/MS	5 years
Purge & Trap	3 years
Gas Chromatography	10 years
High Perf. Liq. Chromatography	3 years
Atomic Absorption	2 years
Sample Preparation	6 years
Data Review	4 years

BILL KEETH, GC/MS Laboratory
Sr. Analyst, Semivolatile Organics
B.S., Chemistry

GC/MS	6 years
Purge & Trap	1 year
Atomic Absorption	4 years

DOUG ANDERSON, GC/MS Laboratory
B.S., Natural Science

Sample Preparation	2 years
GC/MS	1 year

LINDA GODBOLD, GC/MS Laboratory
A.S., Medical Technology

GC/MS	3 years

SANDY GROVENSTEIN, GC/MS Laboratory
B.S., Biology

GC/MS	10 years
Data Review	8 years
Gas Chromatography	14 years
tomic Absorption	2 years
High Performance Liquid Chromatog.	4 years

DIANA HOKE, GC/MS Laboratory
B.A. Chemistry

GC/MS	6 years
Data Review	6 years
Purge & Trap	6 years
Gas Chromatography	6 years
Sample Prep	2 years
Wet Chemistry	1.5 years

SERETHA PAVEY, GC/MS Laboratory
B.A. Mathematics, A.S. Chemistry

GC/MS	1 year

ANN WADE, GC/MS Laboratory
B.S., Environmental Health

GC/MS	3.5 years
Gas Chromatography	6 years
Sample Preparation	4 years
Purge & Trap	3.5 years
Purge & Trap Data Review	3.5 years
Atomic Absorption	1 year

XIANGQUI LANG, GC/MS LABORATORY
High Res Mass Spec Operator
M.S. , Chemistry

High Res GC/MS	6 years
Gas Chromatography	3 years
Purge & Trap	3 years
Data Review	3 years

VICKI L. HALL,
GC Laboratory SectionSupervisor
M.S., Chemistry

ICP	1 year
Gas Chromatography	7 years
High Performance Liq. Chromatography	1 year
Sample Preparation	2 years
Data Review	3 years

PAUL BEEBE, GC Laboratory
B.S., Biology

Sample Preparation	1 year
Gas Chromatography	training

BRETT DEES, GC Laboratory
B.S., Biology/Minor: Chemistry

GC/MS	1 year
Gas Chromatography	3 years
Sample Preparation	1 year
Data Review	2 years

CARL KERKEMEYER, GC Laboratory
B.S., Chemistry

Gas Chromatography	10 years
Sample Preparation	3 years

LISA MALONE, GC Operator
B.A., Poly. Sci.

Gas Chromatography	1 year
Sample Preparation	training
Data Review	1 year

DENISE PETERSON, GC Laboratory
A.S., Environmental Lab Chemistry

Gas Chromatography	4 years
Atomic Absorption	1 year
Sample Preparation	2 years
Data Review	5 years
Inorganic Analysis	1 year
High Perf. Liq. Chromatography	5 years

PHUONG VO, GC Laboratory
B.S., Biology

Gas Chromatography	2 years
High Perf. Liq. Chromatography	2 years

SHAHRIAR "ALI" SHAHREZA, GC Lab
B.S., Biology/Chemistry

Gas Chromatography	2 years
High Performance Liq. Chromatography	4 years
Atomic Absorption	1 year
Sample Preparation	4 years
Data Review	3 years

BRYCE SMITH, GC Laboratory
B.S., Zoology

Sample Preparation	3 years
Data Reveiw	3 years
Inorganic Analysis	1 year
Gas Chromatography	3 years

DESMOND FOSTER,
Extractions Lab Supv.
H.S. Diploma

Sample Preparation	13 years
Inorganic Analysis	5 years

RICKKI AGIMUDIE, Extractions Laboratory
A.S., Drafting

Sample Preparation	4 years

HUAN BUI, Extractions Lab
B.S., Chemistry

Sample Preparation	training
GC/MS	college training
GC	college training

JEFF CUMMINS, Extractions Lab
B.S., Chemistry

Sample Preparation	6 mo.
GC	college training

DAVID LOPER, Extractions Lab
A.S., Accounting

Glassware Prep	2 years
Sample Preparation	1 year

JASON McCASLIN, Extractions Lab
B.S., Biology

Sample Preparation	training

KAYLA RICHERSON, Extractions Lab
H.S. Diploma, College Biology/Zoology

Atomic Absorption	7 years
Sample Preparation	7 years
Data Review	7 years
Inorganic Analysis	7 years
GC/MS	college training
GC	college training

TIM RUTLEDGE, Extractions Lab
H.S. Diploma

ICP	2 years
Gas Chromatography	3 years
Atomic Absorption	4 years
Sample Preparation	10 years
Data Review	5 years
Inorganic Analysis	11 years
High Perf. Liq. Chromatography	2 years

OTTO SIMPSON, Extractions Laboratory
H.S. Diploma

Sample Preparation	3 years

CHRIS STROUSE, Extractions Lab
B. S. Chemistry

Sample Preparation	training

MISSY HAMBY
Lead Sample Custodian
High School Diploma

GC	5 years
Purge & Trap	5 years
Atomic Absorption	2-1/2 years
Sample Preparation	7-1/2 years
Data Review	11-1/2 years
Inorganic Analysis	5 years

KAREN LOLLIS,
Sample Custodian
A.S., Chemistry

Sample Receiving	2 year

Kim Willison
Sample Custodian
High School Diploma

Sample Receiving	1 year

TABLE 1.1 RESUMES

Richard J. Ronan, Ph.D., Laboratory Director

EDUCATION: Ph.D., Inorganic Chemistry
University of Hawaii, 1970

Master of Science, Inorganic Chemistry
University of Hawaii, 1970

Bachelor of Science, Chemistry
Franklin College of Indiana, 1965

PROJECT EXAMPLES:

■ For over 20 years Dr. Ronan has provided technical and management leadership to the environmental community. As principal of ASO Consulting, Inc., he focused these skills to solve problems for senior industrial management and the legal community. Assignments have ranged from project management, document review, and expert witness testimony, to full-time management of the multimillion dollar, multidisciplinary laboratory.

■ Dr. Ronan has over twenty years of government (USEPA) and private sector experience in teaching, basic and applied research, marketing, operations, financial control, client relationships, and major project management. His unique experience has prepared him to handle the integration of all of these areas.

PROFESSIONAL EXPERIENCE SUMMARY:

4/94 to Present
Southwest Laboratory of Oklahoma, Inc., Laboratory Director
As Laboratory Director, Richard Ronan directs the operational functions of all sections of the corporation's largest laboratory, located in Broken Arrow, Oklahoma, including both administrative and analytical. This includes working directly with supervisors and managers from all departments. He is also directly responsible for marketing, business planning and all aspects of the business.

5/90 to 3/94
Principal, ASO Consulting
Experience included support of litigations for six law firms (investigations, depositions, and trials,) a two year business assignment for Huntingdon International Holdings, and other similar assignments. Dr. Ronan designed and developed the QA/QC Program for a $4 billion property owner that oversaw all real estate transactions.

1987 to 1990 **Division Manager and Vice President,** Analytics Div., Roy F. Weston, Inc. Board of Directors (88/90).

1978 to 1987 **Operations Manager and Vice President,** Versar, Inc.

1975 to 1978 **Director Engineering and Research,** Fisher Scientific, Jarrell-Ash Division

1973 to 1975 **Section Chief, Metals,** US EPA Region V, Central Regional Lab, Chicago, IL

1971 to 1975 **Associate Scientist,** Iowa State University, Ames Laboratory

1970 to 1973 **Assistant Professor,** Simpson College, Indianola, IA

Robert W. Harris, Laboratory Manager

EDUCATION: Bachelor of Science Degree, Microbiology
Oklahoma State University, 1971

PROFESSIONAL EXPERIENCE:

1983 to Present
Southwest Laboratory of Oklahoma, Inc.
Laboratory Manager
As the Laboratory Manager of Southwest Laboratory of Oklahoma Mr. Harris directs all aspects of laboratory operation in the Company's newly constructed facility, including scheduling and cost control, staffing, training, customer support, and business development. He holds a B.S. in Microbiology from Oklahoma State University and has over 15 years experience in management of commercial laboratories. Analytical services under his direction include hazardous waste, drinking water and ground water investigation sponsored by a wide variety of governmental and private clients including EPA (e.g.,CLP,RCRA) Corps of Engineers (DERA) and the Air force (OHEL/HSD).

Prior to joining SWL he managed a geochemisty laboratory for Williams Brothers Engineering which supported major oil companies. Mr.Harris has developed supplemental methods for the analysis of hazardous waste when there were no applicable ones available for specific project requirements. His experience in directing the operations of environmental laboratories is exemplified by successful participation in the EPA CLP, U.S. Army Corps of Engineers, and other federal and State certification and accreditation programs. As an analyst Mr. Harris is experienced in the application of analytical methods using SW846, CLP, ASTM, and Standard Methods for organic and inorganic samples.

1981 to 1983 **Assistant Laboratory Director**
Williams Brothers Engineering,
Tulsa, Oklahoma
1975 to 1981—**Senior Research Engineer**

1971 to 1975 **Laboratory Manager**
Kansas City Testing Laboratory,
Kansas City, Missouri;

HARRY M. BORG, Organic Program Mgr.

EDUCATION:
Bachelor of Arts Degree, Chemistry, Minor: Physics, Math
Mount Mary College, Yankton, South Dakota

Additional Training
Hewlett Packard 5988 RTE - 6 System Mgr. Course, 9/97
Hewlett Packard 5987/88/96, Operators Course, 4/86

PROFESSIONAL EXPERIENCE:
2/92 to Present
Southwest Laboratory of Oklahoma, Inc.
Organic Program Manager — Responsible for day to day operations inOrganic department. Oversees performance for all GC/MS operations including extractions, analyses, data review, and data management. Provides technical management for organic EPA contracts. Interviews prospects for organic personnel.

11/89 to 2/92
Laboratory Manager
Conref Labs, Brighton, Colorado — Management of an environmental production laboratory. Responsible for day to day operations and technical management, as well as evaluation and interpretation of analytical results. Responsible as client liaison and for coordination of laboratory projects.

11/88 to 11/89
Technical Services Representative/Environmental Chemistry Manager
Hager Laboratories, Golden, Colorado — Responsible for management of day to day operations in an environmental production laboratory. Technical management, evaluation and interpretation of analytical results. Client liaison and project coordinator.

12/87 to 11/88
Organic Section Supervisor
metaTRACE, Inc., Earth City, Missouri — Responsible for hiring and scheduling of personnel, and Quality control and data review for GC and GC/MS depts.

12/83 to 11/87
Analytical Scientist II
Western Research Institute, Laramie, Wyoming — Responsible for scheduling and training personnel. Analyst in the semivolatile and volatile GC/MS laboratories. Analyses included semivolatile extracts from water, soil, high concentration samples, Pesticide/PCBs by GC and GC/MS, Inorganic analyses of oil shale by-product waters by non-supressed ion chromatography, separation and etection of sulphur anions by non-supressed ion chromatography (HPLC). Responsible for quality control of data. Utilized EPA 600 methods, SW846 methods, RCRA and EPA CLP protocols. Routine maintenance and troubleshooting of the HP59858 and HP5996 GC/MS systems with RTE-6 software and REV D/E.

3/80 to 11/83 **Chemist** — Tosco Corporation, Golden, Colorado & Martinez, Calif. — Responsible for supervision of evening shift personnel during oil-shale project. Coordinated pilot plant ball test program. Coordinated personnel schedule to accommodate research refinery program.

PAUL P. DANIELLO, Assistant Organic Program Mgr.

EDUCATION:
Bachelor of Arts Degree, Biology
Hofstra University, Hampstead, New York, 1977

PROFESSIONAL EXPERIENCE:
5/91 to Present
Southwest Laboratory of Oklahoma
Assistant Organic Program Manager
Responsible for personnel supervision and the daily operations of the Volatiles, BNA, and Dioxins areas. Train operators in the use of instrumentation and data system. Troubleshoot hardware/software problems on GC/MS systems. Review VOA, BNA, and Dioxins/Furans data.

3/88 to 5/91
GC/MS Section Manager
Professional Service Industries/Hall Kimbrell Division, Lawrence, Kansas — Responsible for maintaining three Mass Selective Detectors, an RTE-VI data system, training and supervising operators, scheduling analyses, and final client report production including both electronic and paper deliverables for the USEPA Contract Laboratory Program. Extensive experience in volatile and semivolatile organics, dioxins, furans, pesticides, herbicides, and PCB's in water, soil, air, produce, and other complex matrices. Additionally, supervised the TCLP Zero Headspace Extraction. Responsible for initiating the quality control samples into the laboratory's organic TCLP program.

9/86 to 2/88 **Group Leader** — MetaTrace, Inc., Earth City, Missouri, GC/MS—Responsible for maintenance of six GC/MS units (1-5988, 1-5996, 4-5970), three data systems and the scheduling of sample analysis. Review of Data produced from the six GC/MS Instruments.

5/86 to 8/86 **GC/MS Group Leader** — York Laboratories, Division of YWC, Inc., Whippany, New Jersey, — Operation and maintenance of two GC/MS systems and training of two operators.

6/85 to 5/86 **Organics Department Manager** — Century Laboratories, Inc., Thorofare, New Jersey—oversaw all activities of GC/MS Unit, GC Unit and Sample Preparation Unit. Responsible for properly maintaining nine instruments (3-GC/MS's, 5-GC's, 1-HPLC). Data Review and Report Production.

5/80 to 6/85 **GC/MS Laboratory Supervisor** — H2M Corporation, Melville, New York, Resonsibilities included operation and maintenance of Finnigan OWA and HP-5996 GC/MS systems. Mass Spectral Interpretation of GC/MS Data.

Laboratory Technician—Responsibilities included sample preparation and analysis of trace organic.

Mark Smith, Organic Data Manager

EDUCATION:

Masters of Biomedical Science Program
Major: Pharmacology, 25 credit hrs.
Oral Roberts University, Tulsa, Oklahoma, 1985

Bachelor of Science, Chemistry
Minor, Computer Science
Oral Roberts University, Tulsa, Oklahoma, 1984

PROFESSIONAL EXPERIENCE:

9/91 to Present
Southwest Laboratory of Oklahoma, Inc.
Organics Data Manager
Assembly and review of Organic Data Packages. Development of automation software.

9/89 to 9/91
NET, Inc., Carrollton, Texas
GC/MS Supervisor
Responsible for the scheduling and analysis of all samples for volatiles and semivolatiles by GC/MS. Responsible for the operation and maintenance of four Hewlett-Packard Gc/MS systems: three 5970 MSDs and a 5996. Duties include data review, standards preparation, methods development, SOP writing and updating, writing instrument automation software, employee training, and instrument operation.

11/88 to 9/89
Southwest Laboratory of Oklahoma, Inc., Broken Arrow
Volatile Organics Group Leader
Leader of volatiles GC/MS laboratory. Responsible for the scheduling and analysis of all VOA samples, so that holding time deadlines and turnaround times were met. EPA CLP, 624 and 8240 methods were used. Responsibility to have QA/QC executed at the proper level and frequency. Reviewed data for quality and completeness. Responsible for maintenance of the GC/MS systems.

8/86 to 11/88
Southwest Laboratory of Oklahoma, Inc., Broken Arrow
GC/MS Chemist - Analyzed water, soil, and petroleum based samples for volatiles, semivolatiles, and dioxins in accordance with EPA CLP or SW846 methods and requirements. Wrote procedure files to help automate the analysis of samples and data review. Performed instrument maintenance and repairs.

3/86 to 8/86
Southwest Laboratory of Oklahoma, Inc., Broken Arrow
Extractions and Inorganics Chemist - Prepared sample extracts for semivolatiles, pesticides, PCBs, dioxins, and herbicides per EPA CLP or SW846 requirements. Performed analysis for total organic carbon, oil and grease, chemical oxidation demand, and ion chromatography.

JASON D. RUCKMAN, Inorganic Program Mgr.

EDUCATION: Bachelor of Science, Chemistry
Kansas Wesleyan University, Salina, Kansas, 1985

Additional Studies:
14 hours graduate credit
University of Kansas, Lawrence, Kansas, 1985-86

PROFESSIONAL EXPERIENCE:

6/89 to Present **Southwest Laboratory of Oklahoma, Inc. —Inorganic Program Manager**
Manages and oversees the Contract Laboratory Program (CLP). Reviews data, resolves reporting problems and provides technical assistance to analysts. Directs troubleshooting of instruments to minimize instrument downtime. Performs sample analysis as needed to meet deadline requirements.

3/87 to 6/89 **EPA Metals Manager**
Wilson Laboratories, Salina, Kansas
Primary operator for the Perkin-Elmer 5100. Responsible for analyzing water and soils samples for the EPA according to the 787 contract and assembling CLP packages.

Primary ICP operator (8/86-8/88)
Responsible for everyday maintenance and upkeep of the ICP (Perkin-Elmer 6000).

8/85 to 5/86 **Teaching Assistant, Department of Chemistry**
University of Kansas, Lawrence, KS
Responsible for pre-laboratory lectures, enforcing safety requirements and grading labs and lecture tests.

JOHN WRIGHT, Assistant Inorganic Program Manager

EDUCATION: Bachelor of Science, Chemistry
Northeastern State University, Tahlequah, OK 1984

School of Pharmacy, senior status
Southwestern State University, Weatherford, OK 1986-89

PROFESSIONAL EXPERIENCE:

12/91 to Present **Southwest Laboratory of Oklahoma, Inc. — Assistant Inorganic Program Manager** (12/93 to Present)
Assists the Inorganic Program in overseeing daily operations in the Inorganic Laboratory. Reviews data, resolves reporting problems and provides technical assistance to analysts. Directs troubleshooting of instruments to minimize instrument downtime. Performs sample analysis as needed to meet deadline requirements.

AA Operator (12/91 to 12/93)
Graphite Furnace operation, including all analysis of Pb, Tl, As, and Se. Data review of analyses performed.

2/90 to 12/91 **Analyst III/Group Leader**
National Environmental Testing
— Inorganic analysis and methodology according to EPA protocol. Followed N.E.T. guidelines for quality control. Some cross-training for organics sample preparation for diesels, PAHs, gasolines, and BTXs. Performed analyses for hazardous waste, waste water, and ground water samples. Reviewed data for quality control.

1989 to 1990 **Environmental Chemist**
Environmental Services Company
Performed analytical analyses on waste water, soil, sludge, groundwater, and potable water samples. Also performed analysis for BOD, Cn^{-}, Phenol, $N0_3$—$N0_2$, TKN, N-

NH_3, EPA-WP standards, AOCS standards, and EPA-WS standards. Performed AA analysis using Varian 200

1984 to 1985 **Shift Technician/Analytical Chemist**
Fort Howard Paper Company
Responsible for polymer addition to the kroftas and the resin/caustic system. Tested paper for proper wet strength. Ensured paper met guidelines for quality control. Responsible for paper standards and process. Research to increase productivity involving zeta potentiometry.

CHUCK HOOVER
Quality Assurance Officer

EDUCATION:
Bachelor of Arts Degree in Biology
Minor in Chemistry
Wichita State University, 1976

PROJECT

- Experience with PCB analyses on transformer oils, Hydraulic fluids, soil/sediment, waters and fish flesh.
- Experience in EPA 600 series Methodologies, both extractions and analyses.
- Experience in Trihalomethane Studies on source and drinking water supplies.
- Experience in data review of analyses performed on samples from EPA's Superfund Sites; performed in various laboratories in the EPA's Contract Laboratory Program.
- Metal analysis using Flame/Furnace Atomic Absorption. Extraction/Clean-up of Dioxin/Furans using method 613.

PROFESSIONAL EXPERIENCE:
1987 to Present
Southwest Laboratory
Of Oklahoma, Inc.
QA/QC Officer

1985-1987
Lockheed Engineering and
Management Services
Las Vegas, Nevada, Technical Support Supervisor, Laboratory Performance Monitoring Section.

1983-1985
Southwest Laboratory
Of Oklahoma, Inc.
QA/QC Officer/Extractions Chemist/AA Operator

1982-1983
National Analytical Laboratories, Tulsa, OK
Gas Chromatography/GC/MS Chemist

1980-1982
Williams Brothers Laboratories, Tulsa, OK
Wet Chemistry Section Supervisor

1977-1980
Wichita Water Department,
Water and Waste-water Laboratory,
Wichita, KS
Laboratory Technician

1976-Summer
U.S. Department of Labor
Occupational Health & Safety Administration (OSHA), Kansas City, MO,
Industrial Hygienist (Summer Merit)

Robert A. West,
Computer Services

EDUCATION:
Langston University, Tulsa, OK
Bachelor of Science degree in Computer Science (December 1988)

Coursework:
Computer Management Information Sys.
Software Design and Development
Data Structures and Algorithms II
Database Design

Tulsa Junior College, Tulsa, Oklahoma, Associate degree in Mathematics (May 1986)

PROFESSIONAL EXPERIENCE:

10/90 to Present **Southwest Laboratory of Oklahoma, Inc., Assistant Programmer, Computer Services Department.**

10/88 to 9/90 **Programer Analyst**
Tulsa, Oklahoma
Developed and administrated a UNIX software for retail shelf labeling industry to drive Postscript and Laserjet printers. (ie. NCR Tower, AT&T, Unisys, Intel 386's, IBM AIX, FLEX OS on IBM 4680, SCO Xenix, ALTOS, MS-DOS)

7/88 to 10/88 **Programmer Analyst**
Voice Systems and Services Inc., Mannford
Developed and supported the Voice Mail System software.

6/84 to 7/88 **Data Processing Coordinator**
St. Francis Hospital, Tulsa, Oklahoma
Coordination of Data Processing projects, software product evaluation, created inventory control programs.

TABLE 1.2 LABORATORY CREDENTIALS

PROGRAM PARTICIPATION

USEPA CLP (Contract Laboratory Program)

- Organics Analysis, Multi-Media, Multi Concentration, by GC/MS and GC/EC Techniques. *Note: continuous participation since 1985.*
- Rapid Turn-Around Organics Analysis, Multi-Media, Multi Concentration, by GC/MS and GC/EC Techniques.
- Chemical Analytical Services for Multi-Media, Multi-Concentration Metals and Inorganics.

EMSL Cincinnati, WP/WS
Water Pollution/Water Supply Study Program.

U.S. Army Corps of Engineers, DERP
DERP certification(Defense Environmental Restoration Program).

U.S. Air Force, AFCEE IRP
Installation Restoration Program participant, analytical services for AFB Projects.

U.S. Navy, NEESA CLEAN IRP
Installation Restoration Program participant, analytical services

INEL, (Idaho National Engineering Lab.)
Qualified by ERPSMO to perform analytical services.

HAZWRAP
Certified to perform analytical services for Hazardous Waste Remedial Actions Program.

NRC License
Licensed to perform analysis of coal contaminated waste for environmental pollutants. (License #35-27413-01)

STATE CERTIFICATION

ALABAMA ..... Lab ID #40890
Department of Environmental Management

ARKANSAS ..... Lab ID #N/A
Department of Pollution Control & Ecology

CALIFORNIA ..... Lab ID #1221
Department of Health Services, Env. Lab Program

COLORADO ..... Lab ID #335
Department of Health, Drinking Water Parameters

FLORIDA ..... Lab ID #87326
Department of Health and Rehabilitation

IOWA ..... Lab ID #104
Department of Natural Resources

KANSAS ..... Lab ID #E-1126
Department of Health and Environment

KENTUCKY ..... Lab ID #90065
Department of Environmental Protection

LOUISIANA ..... Lab ID #93-19
Department of Health and Hospitals

MICHIGAN ..... Lab ID #N/A
Department of Public Health

NORTH CAROLINA ..... Lab ID #404
Department of Env. Health & Natural Resources

NORTH DAKOTA ..... Lab ID #R-073
Department of Health and Conservation

OKLAHOMA ..... Lab ID #8728
Water Resources Board

SOUTH CAROLINA ..... Lab ID #79003
Department of Health and Environmental Control

TENNESSEE ..... Lab ID #02929
Department of Health and Environment

UTAH ..... Lab ID #E-117
Department of Health

VIRGINIA ..... Lab ID #00322
Department of Drinking Water

11/93

TABLE 1.3 LABORATORY INSTRUMENTATION & EQUIPMENT INVENTORY

GC/MS Laboratory

DESCRIPTION — **YEAR**

GC/MS Hewlett Packard 5985/87/88 1978
Connected to RTE Aquarius Data System (V)
Packed/Capillary Injector
EI/CI Heated Source
Direct Insertion Probe
Hewlett Packard 5890 Gas Chromatograph
Hewlett Packard 7673 Auto Sampler

GC/MS Hewlett Packard 5995/5996 1982
Connected to RTE Aquarius Data System (VI)
Packed/Capillary Injector
Heated Source
Hewlett Packard 7673 Auto Sampler

GC/MS Hewlett Packard 5970 MSD 1986
Connected to DOS Chemstation with Target data procession on HP710
Packed/Capillary Injector W/ Jet Separator
Hewlett Packard 5890 Gas Chromatograph
Tekmar 2000 with 2016, 2032 Auto Sampler

GC/MS Hewlett Packard 5970 MSD 1987
Connected to DOS Chemstation with Target data procession on HP715a
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Hewlett Packard 7673 Auto Sampler

GC/MS Hewlett Packard 5970 MSD 1988
Connected to DOS Chemstation with Target data procession on HP710
Packed/Capillary Injector W/ Jet Spearator
Hewlett Packard 5890 Gas Chromatograph
Tekmar 2000 with 2016, 2032 Auto Sampler

GC/MS Hewlett Packard 5970 MSD 1988
Connected to RTE Aquarius Data System (VIII)
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Hewlett Packard 7673 Auto Sampler

GC/MS Hewlett Packard 5988 1988
Connected to RTE Aquarius Data System (VI)
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Hewlett Packard 7673 Auto Sampler

GC/MS Hewlett Packard 5970 MSD 1989
Connected to RTE Aquarius Data System (V)
Electronic Pressure Control Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Hewlett Packard 7673 Auto Sampler

GC/MS Hewlett Packard 5970 MSD 1989
Connected to RTE Aquarius Data System (VIII)
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Hewlett Packard 7673 Auto Sampler

GC/MS Hewlett Packard 5970 MSD 1989
Connected to DOS Chemstation with Target data procession on HP715b
Packed/Capillary Injector W/ Jet Spearator
Hewlett Packard 5890 Gas Chromatograph
Tekmar 2000 with 2016, 2032 Auto Sampler

GC/MS Hewlett Packard 5970 MSD 1990
Connected to DOS Chemstation with Target data procession on HP720b
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Hewlett Packard 7673 Auto Sampler

GC/MS Hewlett Packard 5971 MSD 1990
Connected to DOS Chemstation with Target data procession on HP710
Packed/Capillary Injector W/ Jet Spearator
Hewlett Packard 5890 Gas Chromatograph
Tekmar 2000 with 2016, 2032 Auto Sampler

GC/MS Hewlett Packard 5971 MSD 1991
Connected to DOS Chemstation with Target data procession on HP715b
Packed/Capillary Injector W/ Jet Spearator
Hewlett Packard 5890 Gas Chromatograph
Tekmar 2000 with 2016, 2032 Auto Sampler

GC/MS Hewlett Packard 5971 MSD 1991
Connected to DOS Chemstation with Target data procession on HP715b
Packed/Capillary Injector W/ Jet Spearator
Hewlett Packard 5890 Gas Chromatograph
Tekmar 2000 with 2016, 2032 Auto Sampler

12/92

GC/MS Laboratory
(continued)

DESCRIPTION **YEAR**

GC/MS Hewlett Packard 5971 MSD 1991
Connected to DOS Chemstation with Target data procession on HP720b
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Hewlett Packard 7673 Auto Sampler

GC/MS VG Trio-1 1991
Connected to LABBASE data system
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Hewlett Packard 7673 Auto Sampler

GC/MS Hewlett Packard 5970 MSD 1992
Connected to DOS Chemstation with Target data procession on HP715a
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Hewlett Packard 7673 Auto Sampler

GC/MS Hewlett Packard 5972 MSD 1993
Connected to DOS Chemstation with Target data procession on HP720b
Electronic Pressure Control Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Hewlett Packard 7673 Auto Sampler

GC/MS Hewlett Packard 5972 MSD 1993
Connected to DOS Chemstation with Target data procession on HP715b
Packed/Capillary Injector W/ Jet Spearator
Hewlett Packard 5890 Gas Chromatograph
Tekmar 3000 with 2032 Auto Sampler

High Resolution GC/MS VG-70S 1989
Connected to Vax 4000 data system
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Fisons A200S Autosampler

High Resolution GC/MS Autospec 1989
Connected to Vax 4000 data system
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Fisons A200S Autosampler

Data Systems

RTE Data System (VI)
HP A-900 Computer w/Aquarius Rev. F.
HP 7937 H/XP 571 MB Disk Drive
HP 2934 Printer, 200 CPS (3)
HP 9144 16-Track Streaming Tape Drive
HP Ethernet LAN connection
Terminals (4)

RTE Data System (VIII)
HP A-900 Computer w/Aquarius Rev. F.
HP 7937 H/XP 571 MB Disk Drive
HP 3SI Laser Printer, 16ppm
TI Microlaser Printer, 9ppm
HP 9145 32-Track Streaming Tape Drive
HP Ethernet LAN connection
Terminals (3)

Unix Data System (HP720)
HP 735 Workstation w/HP-UX 9.01
Thruputt Maestro Software
48 MB RAM
Quantum 425S 425 MB Disk Drive
Micropolis 1928 2.0 GB Disk Drive
HP 2.0 GB Dat Tape Drive
HP 1.4 MB Floppy Disk Drive
TI Microlaser Printer, 9ppm
19" Color Monitor
17" Samsung X-terminal

Unix Data System (HP710)
HP 710 Workstation w/ HP-UX 8.07
Thruputt Target Software
32 MB RAM
Quantum 425S 425 MB Disk Drive
HP 1.3 GB Disk Drive
HP 4SI Laser Printer, 16ppm
19" Color Monitor
17" Samsung X-terminal

Unix Data System (HP715a)
HP 715/33 Workstation w/ HP-UX 9.01
Thruputt Target Software
32 MB RAM
HP 1.0 GB 3.5" Disk Drive
HP 3SI Laser Printer, 16ppm
19" Color Monitor
17" Samsung X-terminal

GC/MS Laboratory

Data Systems *(continued)*

Unix Data System (HP715b)
HP 715/33 Workstation w/ HP-UX 9.01
Thruputt Target Software
32 MB RAM
HP 1.0 GB 3.5" Disk Drive
HP 2.0 GB external Dat Drive
HP 3SI Laser Printer, 16ppm
19" Color Monitor
17" Samsung X-terminal

Unix Data System (HP720b)
HP 720 Workstation w/ HP-UX 9.01
Thruputt Target Software
32 MB RAM
Quantum 425S 425 MB Disk Drive
Micropolis 1524 1.3 GB Disk Drive
HP 3SI Laser Printer, 16ppm
19" Color Monitor
17" Samsung X-terminal

Support Equipment

Purge and Trap, Tekmar LSC2 (2)
Tekmar, ALS - 10 Position Auto Samplers (2)
Tekmar 1000 - Capillary CRYO Focuser (1)
NesLab Refrigeration Cooler (3)
Ultra Sonic Cleaner, Mettler (1) 1984
Frigidaire Coolers - Extract Storage (2)
PC Designs 80286 Computers
Formsmaster Software (4)
Tekmar LSC 2000 Purge & Trap Units (4)
Tekmar 2016, 16 port ALS's (4)
Tekmar 2032, 16 port ALS's (4)
Tekmar 6016 16 port ALS's
Tekmar 6032 16 port ALS's
Entech 2000 preconcentrator
2016 cm Autosampler for Summa Canisters

GC Laboratory

DESCRIPTION — **YEAR**

Gas Chromatograph 1978
Carle TCD (Nat Gas)

Gas Chromatograph 1978
Carle TCD (Nat Gas)

Gas Chromatograph 1989
Carle TCD (Nat Gas

Gas Chromatograph 1989
Carle TCD (Nat Gas)

Gas Chromatograph 1989
Carle TCD (Nat Gas)

Gas Chromatograph,
VARIAN 3400 ECD/ECD 1985
Packed Capillary Injectors
CTC A200S Auto Sampler

Gas Chromatograph, HP 5890 ECD/ECD ... 1986
Dual Split/Splitless Injectors
Dual HP 7673 Auto Sampler

Gas Chromatograph, HP 5890 ECD/ECD ... 1987
Dual Split/Splitless Injectors
Dual HP 7673 Auto Sampler

Gas Chromatograph, HP 5890 PID/HALL .. 1987
Tekmar LSC2000 Purge and Trap
Tekmar ALS 2016 & 2032 Position Auto Sampler

Gas Chromatograph, HP 5890 FID/NPD 1988
Dual Split/Splitless Injectors
HP 7673 Auto Sampler

Gas Chromatograph, HP 5890 ECD/ECD ... 1988
Dual Split/Splitless Injectors
HP 7673 Auto Sampler

Gas Chromatograph, HP 5890 Series II
ECD/ECD 1989
Dual Split/Splitless Injectors
Dual HP 7673 Auto Sampler

Gas Chromatograph, HP 5890 Series II
ECD/ECD 1989
Dual Split/Splitless Injector
Dual HP 7673 Auto Sampler

Gas Chromatograph, HP 5890 PID 1989
ALS 2016 & ALS 2032

Gas Chromatograph, HP 5890
HALL/PID/FID 1989
Split/Splitless Injectors
HP 7673 Auto Sampler

Gas Chromatograph, HP 5890 HALL/PID .. 1989
Split/Splitless Injectors
HP 7673 Auto Sampler

Gas Chromatograph, HP 5890 PID 1989
Split/Splitless Injectors
HP 7673 Auto Sampler

Gas Chromatograph HP-5890 TCD 1989
Split/Splitless Injectors

Gas Chromatograph HP-5890B ECD 1991
Dual Split/Splitless Injectors
Twin 7673A Auto Samplers

Gas Chromatograph HP-5890 Series II
ECD/ECD 1992
Dual Split/Splitless Injectors
Twin 7673A Auto Samplers
Electronic Pressure Control (EPC)

GC Laboratory
(continued)

COMPUTERS/INTEGRATORS /DATA SYSTEMS

VG 4 Channel Data Systems (4)
Hewlett Packard 3393 Integrators (2)
Hitachi D2000 Chromatographic Integrators (2)
HP 3396 Integrators (2)
486 Computer (2)
386 Computer (5)
286 Computer (5)
Maxima Data System (8 channels)

LIQUID CHROMATOGRAPHY

Kratos HPLC (1)
- Spectroflow 400 Pumps (2)
- Spectroflow 480 Injector (1)
- Spectroflow 783 Programmable Absorbance Detector (1)
- Hitachi 655A - 40 Auto Sampler (1)
- Fluorescence Detector (1)

Waters HPLC (1) 1992
- 715 Ultr Wisp Sample Processor
- 510 HPLC Pump
- 486 Tunable Absorbance Detector

SUPPORT EQUIPMENT

Oven, VWR 1310 (1)
Frigidaire Cooler - Sample Storage (1)

Extractions Laboratory

DESCRIPTION	YEAR
Concentrators	
Centrifuge, International Equip. Co.	1988
Kudera Danish (124) 1000	1987
Dessicator, Boekel	1985
Drying Oven, Blue M	1980
Millipore (24)	1987
Extraction Apparatus	
Continuous Liquid Extractors (102)	1987
Extraction Apparatus	
Soxhlet Extractors (20)	1987
Gel Permeation Clean-up (GPC) Apparatus, ABC	1987
Applied BioSystems UV Detector, Model 757	1988
Gel Permeation Clean-up (GPC) Apparatus, ABC	1991
Sontek UV Detector	1991
Hitachi D2500 Inetgrator	
Varian 4400 Integrator	
Zymark Benchmate	1992
SSI Pumps (2)	
Sontek UV Detectors (2)	
Zymark Turbovap (2)	1992
Marathon GPC	1994
Sontek Pump (1)	
Sontek UV Apparatus (1)	
Delfield Storage Refrigerator, Extracts	
General Electric Storage Refrigerator, Standards	
8-Place Auto Seperatory-funnel Shaker	1988
Glass Columns	
Extraction Apparatus (20) Seperatory Funnels, 2000 ml	
Funnels (75)	
Dioxin Columns (80 sets)	
400 ml Beakers (75)	
250 ml Erlenmeyers (50)	
500 ml Boiling Flask (90)	
Sample Concentrator, Nitrogen Blow Down, 30 Place	1984
Sample Concentrator, Nitrogen Blow Down, 30 Place	1988
Sonic Disruptor, Sonicator (Sonics)	1989
Sonic Disruptor, Sonicator (Sonics)	1990
Sonic Disruptor, Sonicator (Sonics)	1990
Top Loading Balance, Ohaus 400	1992
Top Loading Balance, Ohaus GT 4000	
Knauer UV Photometer	
Kipp & Loven Printer Controller	

Metal Analyses Laboratory

DESCRIPTION	YEAR
Atomic Absorption	
Instrumentation Laboratory Video 22 Double Beam Dual Channel Spectrophotometer with Graphics	1983
TJA Autosampler	
TJA Prep station	
TJA Video 22 Double Beam, Dual Channel Spectro-	
photometer with Graphics	1991
TJA CTF 188	
TJA Autosampler	
TJA Prep Station	
TJA 188 Controlled Temperature Furnace	1991
Perkin Elmer 5100 Zeeman	1990
PE HGA 600	
PE AS-60	
Varian 400Z GFAA (2)	1992
Buck Scientific Mercury Analyzer 400	1990
Buck Scientific Mercury Analyzer 400	1991
Leeman PS200 Mercury Analyzer	1992
Leeman PS200 Mercury Analyzer	1993

Inductively Coupled Plasma

ICP 61 Plasma Spectrometer
(Thermo Jarrel Ash) 1988
Auto sampler-TJA
ARL 3560 ICP 1990
ARL Auto sampler
ICP 61E Trace Analyzer
(Thermo Jarrel Ash) 1993
ICP, TJA Trace Analyzer 1994

Wet Chemistry - Inorganic

DESCRIPTION	YEAR
Air Compressor	1990
Analytical Balance, Mettler H80	1984
Ash Furnace, Lindberg	1984
Balance Analytical, Mettler H80	1984
Balance, toploading, Ohaus model 400	
Balance, heavy duty solution, Ohaus	
Cadmium Reduction Nitrate Columns	1985
Centrifuge, Damon IEC Clinical	1984
Conductivity Meter, HACH	1983
Digestor, 36 place LABCONCO	
Electrode (Ammonia) 95-12 Orion	
Electrode (Fluoride) 94-09-00 Orion	
Flow Injection Analyzer, Lachet Quikchem	1991
Six Place TCLP/EP TOX Tumblers (2)	
Extractors	
Zero head (12)	
Funnels, Buchner	
Millipore Membrane	
Gooch Crucibles	1983
GCA Heater Dessicator/Precision	1984
Glass Dessicators, Large and Small	1983
Glassware	
Heating Mantles	
Electrothermal 500-C (12)	
Hoods, Labconco - 6 Foot	
Hot Plates & Stirrers	1983
Integrator, Hitachi D2000	
Ion Chromatograph, Dionex,	1982
Dionex-DX100 Ion Chromatograph	1992
Karl Fisher Titrimeter, Bechman KF4	1983
Mettler Ultrasonic Cleaner	1982
MuffleFurnace, Lindberg 51828	
Neslab Coolflow CFT-33	1985
Neslab Coolflow CFT-75	1989
Oven, CMS Equatherm D1262	
Oven, Precision	1986
pH Meter, Orion (Portable)	1982
pH Meter, Corning	1983
Plastic Dessicators, Large	1984
Spectrometer, Bausch & Lomb	1984
Spectrometer 21 DB, Bausch & Lomb	
Stack Sampling Equipment, Joy Unit -2	1982
Total Organic Carbon Analyzer, Ionics	1982
Total Organic Carbon Analyzer,	
Schmadzu 5050	1991
Carbon Analyzer Leco CR12	1989
Total Organic Halogens	1983
Dohrman Microcoulometer MCTS-20	
Tumbler (TCLP), Millipore	
Vacuum-Pressure Pump, Thomas	1984
ISCO Autosampler	
CEM Microwave Digestion System-205	
Dionex DX300 Ion Chromatograph	1993

High Hazards Laboratory

DESCRIPTION	YEAR
Analytical Balance, ASP Model 2410	1986
Blender, Waring	1984
Chemical Carcinogen Glove Box,	
LABCONCO	1984
Heated Dessicator, Precision Scientific	1984
Shaker, Eberbach	1984
Special Glassware	
Columbia Fisheries Dioxin Protocol	1985
Sonic Disruptor, Sonicator - Heat Systems	1985
Infrared Spectrophotometer,	
Perkin Elmer (model 337)	
Muffle Furnace, Linberg	
Samsung Storage Refrigerator	
Laboratory Furniture	
1 - Four Foot Hood, KEEWANEE	1985
1 - Six Foot Hood, KEEWANEE	1985

Data Management

DESCRIPTION	YEAR
Computer, Gateway 2000 486/33	1991
16 Mb Memory	
300 Mb Hard Disk	
150 Mb Tape Backup	
450 Watts UPS	
WYSE 60 Terminals (7)	
Epson FX-850 Printers (7)	
EX800 Printers (1)	
LaserJet III Printers (1)	
Computer, Perkin Elmer 3203	1986
LIMS System, 6 Terminals	

Data Management
(continued)

Copiers
Xerox 1065 1989
Xerox 5065 1990
MITA 4055 1987
Savin 7500 1991
MITA DE131
Sharp SF-2027

Facsimiles
Canon FAX-730
Canon FAX 410
Canon FAX L770 1991
Canon FAX L700

Miscellaneous Computer Equipment
GANDALF PACX IV, PBX
Terminal Ports (196)
Computer Ports (96)
INMAC 64 port Smart Switch
Packard Bell 2400 baud modems (4)
Five Hewlett Packard Computers 2117F 1986-1990
2 Megabyte Memory W/RTE 6/VM & Aquarius Rev.E
Disk Drive, HP 7933, 404 Megabyte (5)
Hewlett Packard A900 Computer , 4 mb(3) 1990
Disk Drive, HP 7937, 500 Mb
Tape Drive, HP 9144 (3)
Disk Drive, HP 7935, 404 Megabyte
Disk Drive, HP 7914, 130 Megabyte
Tape Drive, HP 7970B, 9 Track 800 BPI (2)
Tape Drive, HP 7970E, 9 Track 1600 BPI(4)
Terminals, HP2623 (20)
Terminals, HP2648 (2)
Terminals, HP2627 (3)
Terminals, HP2621P (3)
Terminal, HP 2397A (1)
Terminal, HP 2393A (1)
Terminal, HP 2622 (1)
Terminal, HP 2392 (1)
Terminal, HP 150 (1)
Printers, HP2934A (11)
Printer, HP Ruggedwriter (3)
Printer, HP 2563B (1)
One Hundred Computers, IBM PCs and Compatibles 1986-1991
40 to 200 Megabyte Disk Drives
Streamer Tape Back-Up System (2)

Printers 1987-1991
LaserJet, Hewlett Packard Series III (2)
LaserJet, Hewlett Packard Series IIIsi (2)
LaserJet, Hewlett Packard Series II (1)
LaserJet, Hewlett Packard Series IIP (1)
Paint Jet, Hewlett Packard (1)
DeskJet Plus, Hewlett Packard (1)
ScanJet Plus, Hewlett Packard (1)
Epson FX850s (18)
Epson EX1000s (6)
Epson FX100 (1)
Epson FX286 (1)
Epson LQ500 (1)
Epson EX800 (4)
Epson LX800 (1)
Epson EPL-7000 (5)
Epson Action Laser II (4)
Star SG10 (1)
Panasonic KX-P1191 (1)
Panasonic KX P1180 (1)
Panasonic KX-P1092i (1)
Okidata Microline 182 (1)
IBM Proprinter II (1)
Texas Instruments Microlaser (4)

Personnel Training

All positions involve on-the-job training. This training requires the reading of a Standard Operating Procedure; performace of that procedure under close supervision; and documentation of the understanding and proficiency on a form which is part of each departments' SOPs (see figure 1.3).

All training information including method QA/QC as well as health and safety documentation is maintained in the employees' training file. The training files are maintained by the QA/QC section and include resumes, classes taken by the employee, and any certifications obtained by the employee from attending these classes. All refresher training is included in these files also.

GC DEPARTMENT PAGE 1 OF 2

DOCUMENTATION OF QUALIFIED PERSONNEL PERFORMING ANALYTICAL TESTS

ANALYST NAME:	TITLE:	DATE OF HIRE:

TEST CODE:	METHODOLOGY:	ANALYST INITIALS:	SUPERVISOR INITIALS:	DATE APPROVED:
GC110	EPA 601/SW 8010			
GC120	EPA 602/SW 8020			
GC130	SW 8010/8020			
GC140	EPA 601			
GC150	SW 8020			
GC160	EPA 504.1			
GC199	SW 8020			
GC200	SW 846 8330			
GC210	USAE 30			
GC299				
GC300	MOD 8015			
GC310				
GC320	CALIF. GC/FID			
GC330	IOWA GC/FID			
GC400	EPA 610/SW 8100			
GC405	GC/PID			
GC420	SW 8310/HPLC			
GC430	SW 8315			
GC550	EPA 615/SW 8150			
GC560	EPA 615/SW 8150			
GC570	SW 8150			
GC580	EPA 615/SW 8150			
GC599	SW 8150			
GC600	EPA 608/SW 8080			
GC610	SW 8080			
GC620	SW 8080			
GC700	EPA 604			
GC710				
GC720	HPLC			
GC800	EPA 608/SW 8080			

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FIGURE 1.3 Example of Training Documentation

SECTION 2.0 - SAMPLING PROCEDURES
OF
SOUTHWEST LABORATORY of OKLAHOMA, INC.'s
QA/QC MANUAL
HAS BEEN INTENTIONALLY OMITTED
FROM THIS DOCUMENT

3.0 SAMPLE CUSTODY

A critical aspect of sound sample collection and analysis protocols is the maintenance of strict COC procedures. COC procedures include inventorying and documentation during sample collection, shipment, and laboratory processing. A sample is considered to be in an individual's custody if the sample is: (1) in the physical possession or view of the responsible party; (2) secured to prevent tampering; or (3) placed in a restricted area by the responsible party.

3.1 CHAIN-OF-CUSTODY

Sample Label

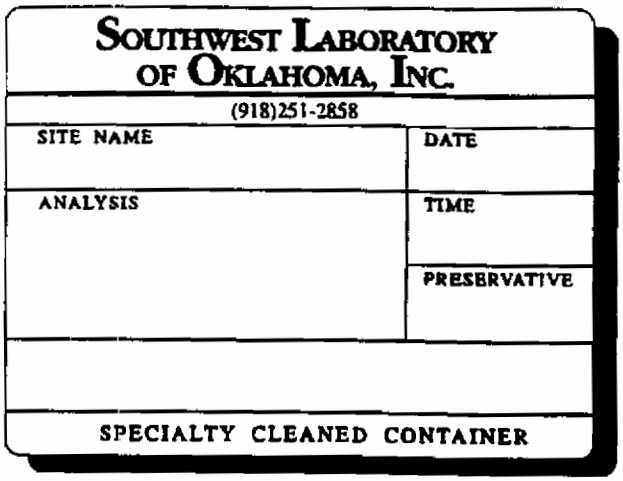
A label is attached to all sample containers at the time of collection. The label is written in indelible ink and contains the following information:

- Sample number/identification
- Date and time collected
- Purpose of the sample (analyte and sample group)
- Source/location and location of the sample
- Contract task number and title of project
- Preservative used (if any)
- Collector's name or initials

An example of a sample label is presented in Figure 3.1.

Chain-of-Custody Record

Sample custody is initiated with the detailed record keeping by the field sampling personnel. COC establishes the documentation and control necessary to identify and trace a sample from sample collection to final analysis. It includes field sample



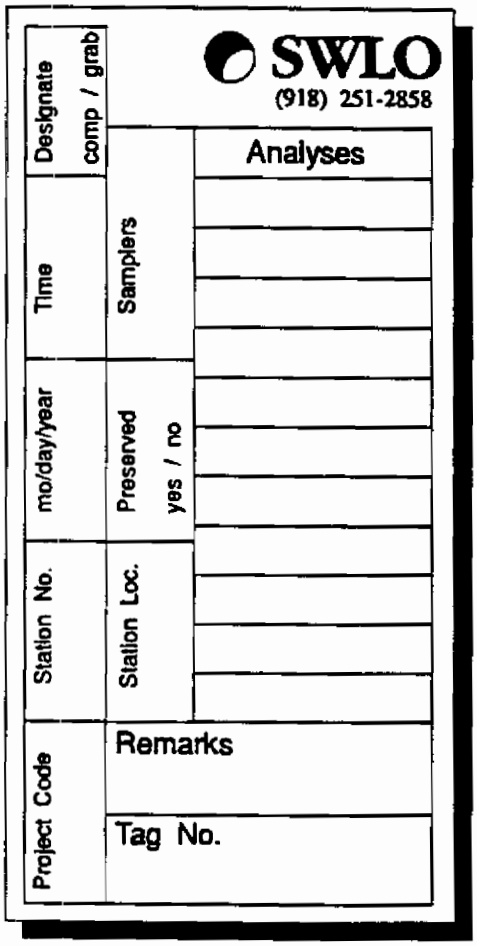


FIG. 3.1 Example of Sample Bottle Label & Sample Tag.

labeling to prevent mix-up, custody seals to prevent sample tampering, secure custody, and provide the recorded support information for potential litigation.

COC forms are used to document the integrity of all samples. To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, a COC form will be filled out for each sample set at each sampling location. The COC form will contain the following information:

- Sample number (for each sample in shipment)
- Collection date (for each sample shipment)
- Time sample was obtained/or collected
- Number of containers of each sample
- Sample description (environmental matrix)
- Analyses required for each sample
- Shipment number
- Shipping address of the laboratory
- Date, time and method of shipment
- Spaces to be signed as custody is transferred.

The individual in charge of shipping samples to the laboratory is also responsible for completing the COC form. This individual will also inspect the form for completeness and accuracy. Any changes made to the COC form shall be initialed by the person making the change. An example of the COC form is presented in Figure 3.2.

Transfer of Custody and Shipment

Samples are to be accompanied by an approved COC record. When the possession of samples is transferred, the individual relinquishing the samples signs and records the date and time on the COC document. The individual receiving the samples repeats the procedure. This record represents the official documentation for all transference of the sample custody until the samples have arrived at the laboratory.

If samples are to be split with another laboratory facility or governmental agency, a separate COC record is prepared for those samples. This COC record indicates with whom the samples have been split and is appropriately signed and dated with the time of transfer of splits.

Laboratory Custody Procedures

The Sample Control program describes the laboratory custody procedures associated with sample receipt, storage, preparation, analysis and security. Sample control is maintained at SWLO through the use of several tracking systems designed to protect sample integrity. Tracking systems include the use of laboratory COC procedures, locked sample storage, sample request forms, and sample analysis requests (in the form of project sheet).

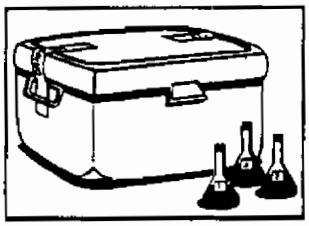
Laboratory COC procedures include sample inventory and record maintenance during sample collection, shipment and laboratory processing. The Sample Custodian (SC) manages and tracks the storage and distribution of samples after their arrival.

An overview of the sample tracking and COC procedure to be employed is presented in the Figure 3.3 flow diagram. It includes the following components:

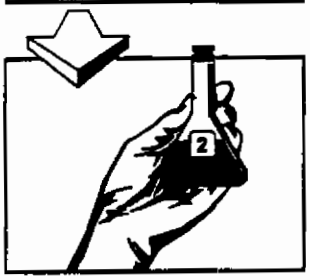
1. Laboratory COC documentation is initiated by the SC when the sample is relinquished by the courier.
2. After sample shipment arrival, the SC begins sample inspection and log-in. Cooler temperatures are recorded for those clients requesting it, otherwise samples received warm will be noted on the Chain-of-custody. Samples are checked for preservation in the sample preparation area and recorded on Extraction/Digestion Logs. All samples are inspected: comparisons are made between the clients paperwork and that paperwork supplied by the Project Officer (i.e., Sample booking Information, Figure 3.5). Anomalies are noted in the COC form and the Client/Lab Communication sheet (Figure 3.6) as the client is notified.

FIG. 3.2 Example of Chain of Custody Record

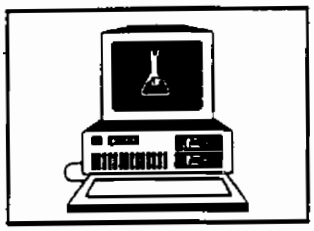
FIG. 3.3 Chain of Custody Sample Tracking Flow Diagram



◀ Sample Receipt

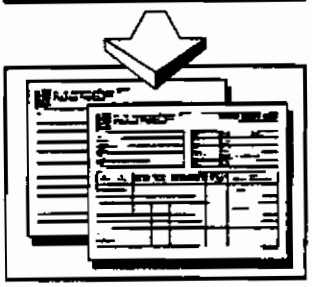


▲ Sample Inspection *(Primary & Secondary Containers)*



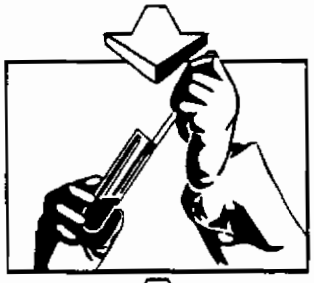
◀ Sample Log-In *(Assigned Unique ID Number)*

Initiate Internal Chain-of Custody Documentation ▶

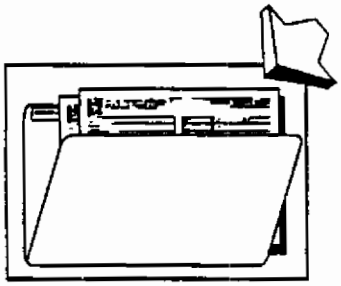


▲ Copy of Document Returned to Appropriate Official(s)

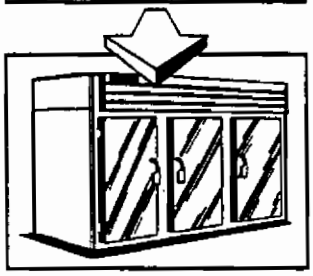
Sample Relinquished to Appropriate Lab Manager *(Sample maintained in locked storage)* ▶



◀ Sample Preparation and Analysis *(Sample maintained in locked storage)*

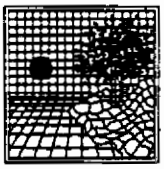


▲ Chain of Custody Document Completed and Filed with Appropriate Data in Contract File



▲ Archive Sample/Extracts and Document Locations in Locked Freezer

▲ Non-essential Sample Remains Disposed of *(Hazardous sample remains incinerated in controlled circumstances, or returned to client.)*



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INITIAL CONTACT FORM

➤ Client:	
➤ Client Contact:	
➤ Client P.O. No:	➤ Date:
➤ Billing Instructions:	➤ Phone No:
	➤ Proposal:
➤ Analytical Requests:	
➤ Method Requests	
➤ Detection Limits:	
➤ Additional QC:	
➤ Number of Samples:	➤ Matrix/Matrices
➤ Shipping Date:	➤ VTSR:
➤ Turn-around Time:	➤ Duration of Project:
➤ Bid Price:	➤ Bid Due Date:
➤ Comments:	
➤ Approximate $ Volume:	
➤ Award Date:	➤ Follow-up Date:
➤ Price:	➤ SWL Contact:
➤ Additional Information:	
➤ Required Qualifications:	
➤ Decision Criteria:	
➤ Other Bidders:	
➤ Data Package:	

FIG. 3.4 Project Sheet (Initial Contact Form)

3. Each sample is assigned a unique SWLO laboratory identification number which is cross-coded with the client's identification. Sample identification information is entered into the computerized laboratory data base, and the assigned number is used to track sample locations and status throughout the analytical process.
The following sample information is recorded into the computerized laboratory data base system:

   - Customer and project information
   - Date of receipt
   - Client identification
   - Date sampled
   - Matrix Type
   - Number of containers
   - Analytical requirements
   - Other pertinent comments

4. The SC logs in samples with the tests and test code information supplied by the project officer (figure 3.5 & 3.7)

5. The field COC document is completed and copies are returned to the appropriate party(s).

6. After the sample is logged in, a LIMS generated work sheet is generated (Figure 3.9).

7. The work sheet informs the analysts/departments of samples in-house.

8. This internal sample tracking sheet documents the movement of the sample from storage to sample preparation and back to sample storage.

9. While within the laboratory, sample integrity is maintained through the use of locked storage areas. Samples remain in locked storage areas except when being analyzed.

10. Based on specific contract requirements, any remaining samples are either archived in locked storage areas or disposed of properly. (See figure 3.14)

In addition to the internal and external COC documents, a computer-generated listing of the sample analysis parameters is used to control sample flow and facilitate tracking within the laboratory. Each laboratory unit is given the list of parameters and is responsible for maintaining sample integrity (holding time), fulfilling COC requirements, scheduling sample flow, and tracking sample status.

3.2 PROJECT INITIALIZATION

Sample Custodian

SC for the laboratory has duties and responsibilities that include but are not limited to:

- Receiving samples
- Inspecting sample shipping containers for presence, absence, and condition of:

  Custody seals, locks, "evidence tapes", etc.

  Container breakage and/or container integrity

- Recording the condition of both shipping containers and sample containers (bottles, jars, cans, etc.) on appropriate forms
- Signing appropriate documents shipped with samples (i.e., airbills, COC record(s), traffic reports, etc.)
- Verifying and recording agreement or non-agreement of information on sample documents (i.e., sample tags, COC records, traffic reports, airbills, etc.) on appropriate forms; if there is a variance, the Project Officer (PO) is notified immediately
- Initiating the paperwork for sample analyses on appropriate laboratory documents
- Labeling samples with laboratory sample numbers and cross-referencing laboratory numbers with client numbers and sample tags (Figure 3.8.)

- Placing samples, sample extracts, and spent samples into appropriate storage and/or secure areas

- Controlling access to samples in storage and assuring that laboratory standard operating procedures are followed during sample movement

- Monitoring sample COC in the laboratory

- Monitoring sample tags

- Monitoring storage conditions for proper sample preservation

- Returning shipping containers to sampling teams or clients.

3.3 SAMPLE RECEIPT

Upon arrival at the laboratory, all sample shipping containers are opened and inspected. Field sampling personnel are notified on the same day of any problems concerning the samples or documents associated with the shipment. If samples arrive on a Saturday and field sampling personnel are unavailable, notification is made on the next working day.

Initial Receipt

All samples will be received by one of the Sample Custodians (SC). For weekend sample receipt, a designated person shall receive the samples and store them properly for sample log-in processing the next business day.

All samples received shall be considered to be hazardous samples, and all shipping containers shall be opened in an approved exhaust hood or an approved, well-ventilated area. All personnel associated with sample receipt are required to become familiar with safety procedures for the handling of hazardous samples.

The objective of the sample receipt procedure is to ensure that all pertinent information about the condition of the sample is recorded.

Examination of Shipping Container

The SC or will examine the shipping container and shall record the following information on the COC sample log-in sheet. Only one project or sample batch may be recorded per sheet.

- Condition of container, noting any damage, etc.

- Presence/absence of COC seals and their condition

- Labeling on shipping container

Opening Shipping Container

No shipping containers should be opened except under an approved hood or in an approved, well-ventilated area. Approved hood space and/or approved, well ventilated areas shall be determined by the Laboratory's Health and Safety Officer or the Corporate Health and Safety Officer. Prior to the removal of samples, plastic-backed absorbent pads should be laid out to receive sample bottles. The SC shall note on the sample log-in form the following:

- Presence/absence of the COC record(s)

- Presence/absence of airbills and/or bills of lading documenting shipment of samples

- Necessary project and sample information enclosed with the shipment

- When samples arrive without ice or blue ice.

Sample Removal

The SC shall note on the COC form the following:

- Condition of samples (intact, broken, leaking, cold or ambient, headspace in VOA vials, etc.)

- Presence/absence of sample tags

If the sample tags are present:

- Record sample tag document control numbers

- Compare sample tags with COC record(s)
- Document whether these numbers agree
- If the sample tags are not listed on the COC record, record this fact.

If an odor is noticed after opening the shipping container prior to sample removal, it must be noted on the COC.

Sample Document Verification

The SC will compare the following documents to verify agreement among the information contained on them: (a) COC; (b) sample tags; (c) prepared project sheets; and (d) contract requirements. The SC shall document agreement among the forms and shall note any discrepancies found on the sample log-in sheet.

- If all samples recorded on the COC record were received and no problems observed, the SC will sign the COC record in the "received for laboratory by" box.
- If problems are noted, the SC will sign the COC record and note problems in the "remarks" box or reference another form that details the problems.
- If discrepancies are found, they shall be reported to the PO for clarification.

In addition, samples to be analyzed are checked for holding time requirements as listed in the Tables 2.1 and 2.2. Where sample preservation and/or sample holding time requirements are not in accordance with the table, the appropriate PO is notified.

3.4 SAMPLE LOG-IN

Following inspection of shipping containers, records, and samples, the sample information will be added to the project information on the project sheet by the SC. Should any of the project information be incomplete or any other problems arise, the sample shall be placed on hold and the problems will be noted on the Client/Laboratory Communication System Sheet (Figure 3.6). This problem sheet shall be forwarded to the PO for resolution. The samples shall remain on hold until all information necessary for log-in is received. Information concerning the sample will be entered into the laboratory data base to maintain an official record of receipt of the sample. Corrections will be made once the PO resolves all problems.

Any samples not properly preserved will be noted on the COC and the field sampling manager will be notified immediately of the problem. He will determine the necessary corrective action.

In the event holding times may be exceeded, the PO shall contact the field sampling manager or the client immediately to correct any log-in problems. In the event holding time for analysis is exceeded, the PO or SC shall notify the field sampling manager or the client and request either resampling or instruction for proceding with the analysis. If analysis is to be performed on the original samples, the PO shall note the fact that the holding time has been exceeded, and a comment to this effect shall be added to the final report.

3.5 SAMPLE SPLITTING

When clients supply their own containers or when bulk samples are received, the SC shall split the samples to provide sufficient aliquots for each analytical procedure that is to be performed. The following guidelines shall be used to determine the manner in which samples are split.

Water samples - Inorganic Parameters Only

The SC with the appropriate laboratory section manager determine the minimum sample quantities required for analysis. If insufficient sample exists to produce the aliquots needed, the SC shall contact the PO for a priority list of parameters, shall split the samples into proper containers, and shall complete the log-in procedure.

Water Samples - Organic and Inorganic Parameters

When bulk samples arrive for both inorganic and organic analysis, the SC or personnel doing volatile analyses shall split out a portion for any required volatile analysis and transfer the samples

CLIENT BOOKING SHEET

QA/QC DEPARTMENT

DATE OF CALL:_____ CLIENT:_____

CLIENT CONTACT:_____ PROJECT:_____

SHIP DATES:_____ REQUIRED TAT:_____

DELIVERABLES: CLP ☐ TAB—QC ☐ SPECIAL ☐ CONTRACT:_____

MS DEPARTMENT

VOA	BNA	DIOXIN	OTHER
W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐

GC DEPARTMENT

PEST/PCB	HERB	PAH	EXPLOS.	OTHER
W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐

INORGANIC DEPARTMENT

ICP	FURNACE	Hg-CV	CN	WET CHEM.
W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐

AATS

BTEX	TPH MOD-8015	TPH 418.1	O&G	OTHER
W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐

TEST CODES/COMMENTS:_____

_____

_____

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FIG. 3.5 Client Booking Sheet

CLIENT/LABORATORY COMMUNICATION SYSTEM

PROJECT OFFICERS DEPARTMENT

SWL_____/AATS_____

TELEPHONE RECORD LOG

➤ In Reference to Case Contract/Proposal:

➤ Date of Call:

➤ Client Name:

➤ Client Contact:

➤ Call Initiated By: ❒ Client ❒ Laboratory

➤ In reference to data for the following sample number(s):

➤ Sumary of Questions/Issues Discussed:

➤ Summary of Resolution:

Signature: *Date:*

➤ Distribution: ❒ Lab Copy ❒ Client Copy ❒ Project Officer Copy

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FIG. 3.6 Client/Laboratory Communication System

ANALYTICAL REQUEST FORM

SAMPLE RECEIVING DEPARTMENT

➤ Client:	➤ Code:
➤ Client Contact:	➤ Phone No.:
➤ Project Name:	➤ P.O. #:
➤ Location:.	➤ Receipt Date:
➤ Deliverables:	

ANALYTICAL REQUESTS

Sample ID	Quantity.	Matrix	Code	Code	Code	Code	TAT

➤ Special Provisions

➤ Reporting Address:

➤ Billing Address:

➤ SWL Contact: ➤ Date Prepared:

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FIG. 3.7 Sample Request Form/Analysis Request Form

FIG. 3.8 Sample Log-In Sheet

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1700 WEST ALBANY • BROKEN ARROW, OK 74012-1421
OFFICE (918) 251-2858 • FAX (918) 251-2599

SAMPLE LOG-IN SHEET

SAMPLE RECEIVING DEPARTMENT

Date:	
Custodian: *(Signature)*	
Document Control #:	
Case Number:	
Airbill Number:	

CUSTODY SEAL:	☐ Present ☐ Absent	☐ Intact ☐ Not Intact
CHAIN OF CUSTODY:	☐ Present ☐ Absent	
SAMPLE TAGS:	☐ Present ☐ Absent	
SAMPLE TAG NUMBERS:	☐ Listed ☐ Not Listed	(On Chain-of-Custody)
SMO FORMS:	☐ Present ☐ Absent	

Date Received	Time Received	Chain-of-Custody Record Number	SMO Sample Numbers	Corresponding Sample Tag Nos.	Corresponding Assigned Lab Nos	Does Info. on Custody Rec, Traffic Rept. and Sample Tags Agree?	REMARKS (Condition of Sample Shipment, etc.)

[SR005-0492-01]

SAMPLE LOG-IN SHEET

Lab Name: _____ Page ____ of ____

Received By (Print Name): _____ Log-in Date: _____

Received By: (Signature): _____

Case Number: _____

Sample Delivery Group No: _____

SAS Number: _____

REMARKS:

1. Custody Seal(s) — Present/Absent* Intact/Broken
2. Custody Seal Nos.: _____ _____
3. Chain-of-Custody Records — Present/Absent*
4. Traffic Reports or Packing List — Present/Absent
5. Airbill — Airbill/Sticker Present/Absent*
5. Airbill No: _____ _____
7. Sample Tags — Present/Absent
   Sample Tag Numbers — Listed/Not Listed on Chain-of-Custody
8. Sample Condition: — Intact/Broken*/ Leaking
9. Does information on custody records, traffic reports, and sample tags agree? — Yes/No*
10. Date Received at Lab: _____
11. Time Received _____

Sample Transfer

Fraction: _____ _____

Area #: _____ _____

By: _____ _____

On: _____ _____

	CORRESPONDING		
EPA SAMPLE #	SAMPLE TAG #	ASSIGNED LAB #	REMARKS: CONDITION OF SAMPLE SHIPMENT, ETC.

* Contact SMO and attach record of resolution

Received By: _____

Date: _____

Logbook No: _____

Logbook Page No: _____

FORM DC-1

3/90

FIG. 3.8 (cont.) EPA-CLP Sample Log-In Sheet

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 W. ALBANY SUITE C
BROKEN ARROW, OK 74012-1421

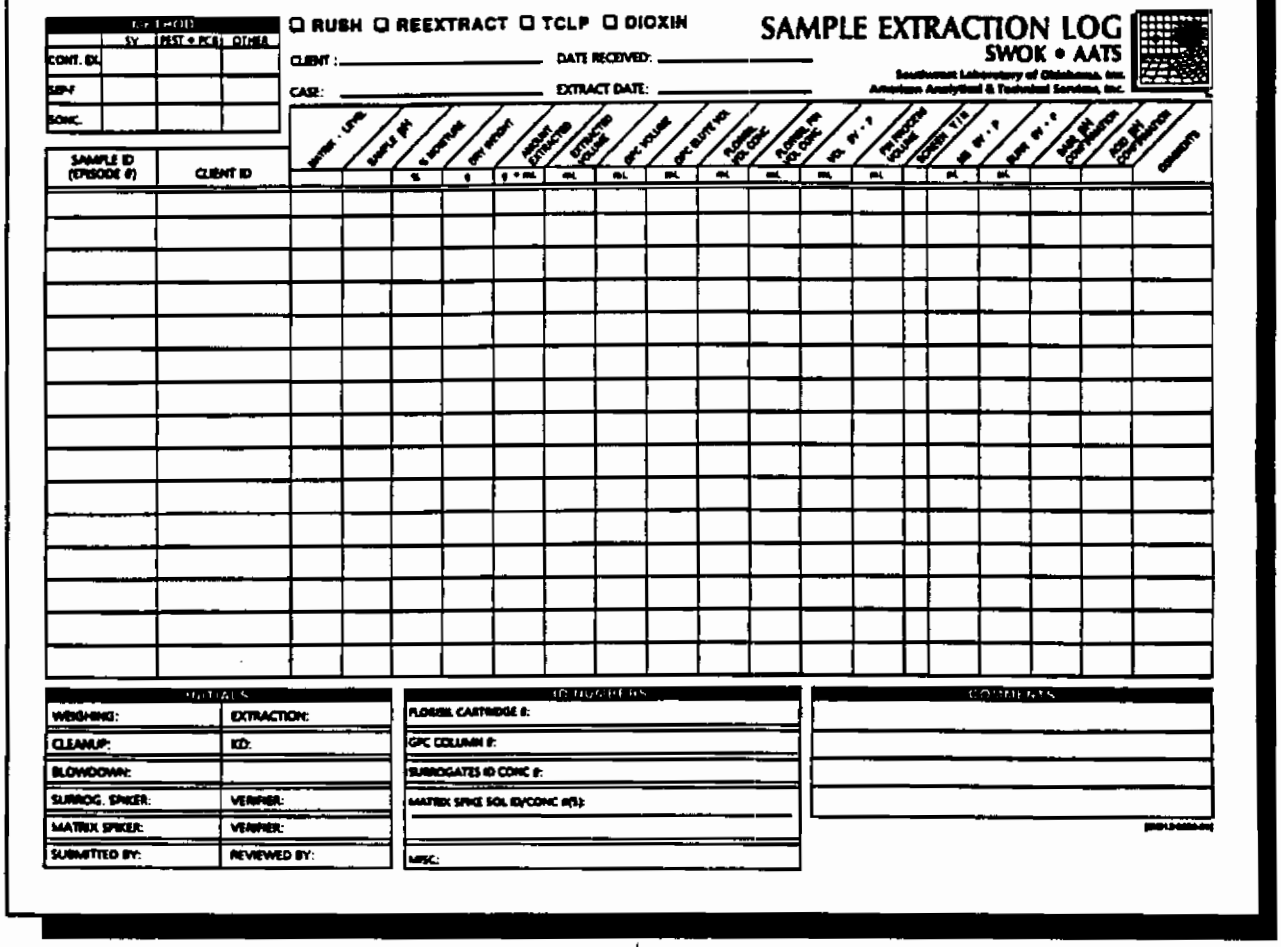
Date: 10/27/93
Episode: 16075
Client: EPA
Project: 21032

SAMPLE LOG-IN RECORD

SAMPLE #	DATE IN	DESCRIPTION	SDG	MA	MC	TEST	PRI	DUE	CONTAINER	DESCRIPTION	RESULTS	ANALYST	DATE/TIME
16075.01	10/25/93	FT919 10A/BC		W	18	GC815	4	11/14/93	MMOPOR	PEST CLP OLM016			
										EXTRACTION		MT	10/26/93
						MS315	4	11/14/93	ABCDEF	VOA-CLP OLM01.8	_____	_____	_____
						MS515	4	11/14/93	GHIJKL	BNA CLP OLM01.8			
										EXTRACTION		TB	10/25/93
16075.02	10/25/93	FT920 (CASE#21032)		W	6	GC815	4	11/14/93	EF	PEST CLP OLM016			
										EXTRACTION		MT	10/26/93
						MS315	4	11/14/93	AB	VOA-CLP OLM01.8	_____	_____	_____
						MS515	4	11/14/93	CD	BNA CLP OLM01.8			
										EXTRACTION		TB	10/25/93
16075.03	10/25/93	FT921 (CASE#21032)		W	6	GC815	4	11/14/93	EF	PEST CLP OLM016			
										EXTRACTION		MT	10/26/93
						MS315	4	11/14/93	AB	VOA-CLP OLM01.8	_____	_____	_____
						MS515	4	11/14/93	CD	BNA CLP OLM01.8			
										EXTRACTION		TB	10/25/93
16075.04	10/25/93	FT922 (CASE#21032)		W	6	GC815	4	11/14/93	EF	PEST CLP OLM016			
										EXTRACTION		MT	10/26/93
						MS315	4	11/14/93	AB	VOA-CLP OLM01.8	_____	_____	_____
						MS515	4	11/14/93	CD	BNA CLP OLM01.8			
										EXTRACTION		TB	10/25/93

Total samples = 4

FIG. 3.9 Sample Work Sheet



SAMPLE EXTRACTION LOG

SWOK • AATS

Southwest Laboratory of Oklahoma, Inc.
American Analytical & Technical Services, Inc.

☐ RUSH ☐ REEXTRACT ☐ TCLP ☐ DIOXIN

CLIENT: _____ DATE RECEIVED: _____

CASE: _____ EXTRACT DATE: _____

METHOD	SV	PEST + PCB	OTHER
CONT. ID:			
SERV			
SONIC.			

SAMPLE ID (EPISODE #)	CLIENT ID	MATRIX - LEVEL	SAMPLE pH	% MOISTURE	DRY WEIGHT	AMOUNT EXTRACTED	EXTRACTED VOLUME	GPC VOLUME	GPC ELUTE VOL	FLORISIL VOL CONC	FLORISIL PCB VOL CONC	VOL IN - F	FM FACTOR VOLUME	SCREEN T/S	VOL IN - F	FINAL IN - F	BASE pH CONFIRMATION	ACID pH CONFIRMATION	COMMENTS
				%	g	g • mL	mL	mL	mL	mL	mL	mL	mL	µL	mL	mL			

STATUS

WEIGHING:	EXTRACTION:
CLEANUP:	ID:
BLOWDOWN:	
SURROG. SPIKER:	VERIFIER:
MATRIX SPIKER:	VERIFIER:
SUBMITTED BY:	REVIEWED BY:

ID NUMBERS

FLORISIL CARTRIDGE #:

GPC COLUMN #:

SURROGATES ID CONC #:

MATRIX SPIKE SOL ID/CONC #(S):

MISC:

COMMENTS

FIG. 3.10 Internal SampleTracking (Extraction Log)

VOLATILES GC/MS RUN LOG

VOLATILES GC/MS DEPARTMENT

SWOK / AATS

TUNE FILE ID: _____ METHOD FILES _____

INSTRUMENT: _____ DATE _____

CB/ID FILE ID: _____ ARCHIVE TAPE #: _____

Time	File	SWOK/AATS Sample ID	Client ID	Vol.	IS Area	Surr. Rec.	Lin. Chk.	Comments	OK/ RR

Analyst Signature

SWL/AATS • 1700 West Albany • Broken Arrow, Ok 74012 [MGM008-0492-01]

FIG. 3.11 Internal SampleTracking (Volatiles GC/MS Run Log)

SWLO / AATS Sample Digestion Record

Date : 04/05/1994 Batch: 04059411 Analyst: TG
Type : MT600 ACID DIGESTION - ICP/AA Client: CLP-PRI MEM-TX Matrix: W

LAB ID	Client ID	TYP	Initial Weight		Final Weight		Color Before	Color After	Texture	pH	Comments
PBW		LB	100	ML	100	ML					
LCSW		BS	100	ML	100	ML					1.0 ML W-1,W-2,85
17824.01	940308D00275		50	ML	50	ML	CO/CL	CO/CL		<2	
17824.01MS	940308D00275		50	ML	50	ML	CO/CL	CO/CL		<2	0.5 ML W-1,W-2,85
17824.01MSD	940308D00275		50	ML	50	ML	CO/CL	CO/CL		<2	0.5 ML W-1,W-2,85
17824.02	940309A05370		100	ML	100	ML	CO/CL	CO/CL		<2	
17824.03	940309A05378		100	ML	100	ML	CO/CL	CO/CL		<2	
17824.04	940309A05376		100	ML	100	ML	CO/CL	CO/CL		<2	
17836.01	940309D00282		100	ML	100	ML	CO/CL	CO/CL		<2	
17836.02	940309D00283		100	ML	100	ML	CO/CL	CO/CL		<2	
17836.02MS	940309D00283		100	ML	100	ML	CO/CL	CO/CL		<2	1.0 ML W-1,W-2,85
17836.02MSD	940309D00283		100	ML	100	ML	CO/CL	CO/CL		<2	1.0 ML W-1,W-2,85
17836.03	940310D00284		100	ML	100	ML	CO/CL	CO/CL		<2	
17836.04	940311A05412		100	ML	100	ML	CO/CL	CO/CL		<2	
17836.05	940311A05413		100	ML	100	ML	CO/CL	CO/CL		<2	
17836.06	940311A05415		100	ML	100	ML	CO/CL	CO/CL		<2	
17836.07	940310A05394		100	ML	100	ML	CO/CL	CO/CL		<2	
17836.08	940311A05414		100	ML	100	ML	CO/CL	CO/CL		<2	
17860.01	940314D00285		50	ML	50	ML	CO/CL	CO/CL		<2	
17860.01MS	940314D00285		50	ML	50	ML	CO/CL	CO/CL		<2	0.5 ML W-1,W-2,85
17860.01MSD	940314D00285		50	ML	50	ML	CO/CL	CO/CL		<2	0.5 ML W-1,W-2,85
17860.02	940314D00286		100	ML	100	ML	CO/CL	CO/CL		<2	
17860.03	940314D00287		100	ML	100	ML	CO/CL	CO/CL		<2	

FIG. 3.12 Internal SampleTracking (Sample Digestion Record)

SAMPLE DISTILLATION RECORD

PAGE ____ OF BOOK ____

INORGANIC DEPARTMENT

Distillation Date _____ Distillation Batch No. _____ Analyst _____

Type of Analysis _____ Client _____ Matrix _____

Sample ID	Client ID	Initial wt/vol (g/mL)	Final Vol. (mL)	Description (Water color, Clarity, Soil color, Texture Artifacts)	pH	Sulfides Present (Y or N)	Chlorine Present (Y or N)	Comments (Spiking Solution)

FIG. 3.13 Internal SampleTracking (Sample Distillation Record)

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

SAMPLES READY TO BE ARCHIVED

PRODUCED ON 04/04/91

SAMPLE	CLIENT	DESCRIPTION	MATRIX	REPORTED
5447.08	RS&A	MW-2	W	04/03/91
5447.09	RS&A	MW-21A	W	04/03/91
5447.10	RS&A	MW-21B	W	04/03/91
5447.11	RS&A	MW 18/B	W	04/03/91
5447.12	RS&A	MW-3B	W	04/03/91
5447.13	RS&A	MW-5	W	04/03/91
5447.14	RS&A	MW-3	W	04/03/91
5447.15	RS&A	MW-4	W	04/03/91
5447.16	RS&A	MW-14	W	04/03/91
5447.17	RS&A	MW-2aB	W	04/03/91
5447.18	RS&A	MW-15A	W	04/03/91
5447.19	RS&A	MW-25	W	04/03/91
5447.20	RS&A	MW-34	W	04/03/91
5447.21	RS&A	TRIP BLANK	W	04/03/91
5447.22	RS&A	MW-6	W	04/03/91
5447.23	RS&A	MW-24	W	04/03/91
5447.24	RS&A	MW-23	W	04/03/91
5447.25	RS&A	MW-22A	W	04/03/91
5447.26	RS&A	MW-18B	W	04/03/91
5447.27	RS&A	MW-11B	W	04/03/91
5447.28	RS&A	MW-29	W	04/03/91
5447.29	RS&A	MW-33	W	04/03/91
5447.30	RS&A	MW-32	W	04/03/91
5447.31	RS&A	MW-30	W	04/03/91
5447.32	RS&A	EQUIP BLANK	W	04/03/91
5447.33	RS&A	MW-11A	W	04/03/91
5447.34	RS&A	MW-18T	W	04/03/91
5447.35	RS&A	MW-19	W	04/03/91
5447.36	RS&A	MW-20	W	04/03/91
5447.37	RS&A	MW-27	W	04/03/91
5447.38	RS&A	MW-28	W	04/03/91
5447.39	RS&A	TRIP BLANK	W	04/03/91
5450.01	KIMCLARK	OUTFALL001	W	04/01/91
5452.01	FHC	5-1	S	04/03/91
5452.02	FHC	5-2	S	04/03/91
5452.03	FHC	5-3	S	04/03/91
5452.04	FHC	5-4	S	04/03/91
5452.05	FHC	5-5	S	04/03/91
5452.06	FHC	6-1	S	04/03/91
5452.07	FHC	6-2	S	04/03/91
5452.08	FHC	6-3	S	04/03/91
5452.09	FHC	6-4	S	04/03/91
5452.10	FHC	6-5	S	04/03/91
5461.01	RS&ASSOC	5211.01	W	04/01/91
5466.01	ATAS	1788.01	S	04/02/91
5466.02	ATAS	1788.02	S	04/02/91
5466.03	ATAS	1788.03	S	04/02/91
5466.04	ATAS	1788.04	S	04/02/91
5467.01	ATAS	1789.16	S	04/03/91

FIG. 3.14 Sample Archive Record Sheet

to the Process Laboratory where the remainder of the organic split shall be made by the Organic Extraction Group Leader. The sample will be returned to the SC to complete samples splitting for the inorganic portion, as described above.

Sediments/Soil Samples

Every effort will be made to acquire duplicate aliquots of these matrices. The sample will be made homogeneous after any portion required for volatile analysis has been removed by one or all of the following procedures:

- Stirring
- Air drying and grinding
- Particle separation
- Quartering

The Organic Extraction Group Leader shall obtain the aliquot for organic analysis after any portion required for volatile analysis has been removed. The remainder shall be given to the Inorganic Laboratory Supervisor for further splitting. If insufficient material exists to provide minimum quantities, the PO shall provide a priority list of parameters.

3.6 SAMPLE SECURITY

Samples are in locked storage areas except during laboratory analysis. The work sheet informs the analyst of what samples are needed for sample preparation and/or analysis (Figure 3.8). Internal custody of the sample is documented in the Sample Digestion/Extraction Logs, or in the case of Volatiles, the GC/MS Run Log (see Figure 3.9 through Figure 3.12.) All laboratory personnel who receive samples are responsible for the care and custody of samples from the time each sample is received until samples (or appropriate documentation as to disposition of the empty containers) are returned to storage. All subsets (extraction, digestates, etc.) of the samples shall be kept in locked storage which is controlled by the appropriate laboratory manager. (See Appendix D, Standard Operating Procedure for Laboratory and Sample Security.)

3.7 SAMPLE STORAGE AND DISPOSAL

Once the samples have been logged into the computer system, the SC shall be responsible for the following:

Sample Storage

1. Samples and extracts shall be stored in a secure area.
2. Samples shall be removed from the shipping container and stored in their original containers unless damaged.
3. Damaged samples are to be documented and PO or the client is contacted immediately to notify him of the damaged samples.
4. Storage area is to be kept secured at all times. SC will control access to the storage area.
5. Samples removed from storage will be documented. All transfers of samples are documented in the internal COC.
6. VOA samples will be stored separately from other samples.
7. Standards are not stored with samples.

Sample Disposal

1. Upon completion of the analysis, an archive list is generated each month (Figure 3.14).
2. When sample analysis and all QC checks have been completed and a final report has been issued, the unused sample portion shall be archived for a period of not less than 60 days after the report has been issued.
3. The SC shall be responsible for returning all unused bottles, shipping containers, packing materials, blue ice packs, and if requested, the unused sample portions to the client.
4. Any sample remains shall be properly disposed of after a 120-day period, unless further instructions are received from the PO or cli-

ent. Sample disposal shall be documented on the archive list (Figure 3.14.)

5. For more information (Details on Disposal of Samples & Waste) see Appendix D-25.

3.8 FIELD DOCUMENTATION

Field Sample Identification

Sample tracking is accomplished in the field by assigning each sample a unique number as it is collected. This number is traceable back to the day, time, site, and depth (where appropriate) of collection. This is recorded on a sample label and the COC form as well as in the field logbook. All containers are labeled prior to actual sampling.

Daily Logs

Daily logs are kept during field activities by the Field Supervisor at each site. These daily logs are kept in a bound field notebook of water-resistant paper. All entries are made legibly in indelible ink, signed, and dated. Information that is to be recorded in the field notebook includes:

- Date, time, and place of sampling
- Field QC samples, as applicable
- Weather conditions at time of sampling, including ambient temperature and approximate wind direction and speed
- Data from field analyses (e.g., temperature, specific conductance, pH, and alkalinity of water samples)
- Turbidity of water samples
- Data from physical tests (sludge tests, etc.)
- Observations about site and samples (odors, appearance, etc.)
- Information about any activities, extraneous to sampling activities, that may affect the integrity of the samples (such as low-flying aircraft nearby, fossil-fueled motors being used nearby, painting operations being carried out upwind of sampling sites, etc.)
- Analyses and required preservation techniques
- Sample cooler temperature readings.

Corrections to Documentation

When it becomes necessary to make corrections to any form of documentation (e.g., sample tags, COC forms, daily logbooks), the obsolete information is crossed out with a single line and the changes are made and initialed by the person making the change.

Disposition of Documentation

Upon conclusion of the field effort at a sampling site, all field documentation (i.e., maps, well logs, logbooks, photographs) is clearly labeled and placed in the project files.

3.9 LABORATORY DOCUMENTATION

Samples Labeling/Identification

The SC shall assign laboratory sample numbers. These numbers will be listed on a document to cross-reference with the client number, sample tag number, and laboratory sample number.

Laboratory sample numbering is comprised of an "Episode" number (Episode meaning client batch) followed by .01 for the first sample, .02 designating second sample, etc. These numbers are sequentially generated by the SWLO "LIMS" system.

	SWLO ID	Client ID
Example:	3001.01	Sample A
	3001.02	Sample B
	3001.03	Sample C

This unique sample number shall be used for sample identification during storage, analysis and data reduction, data validation, and reporting.

Filling of Log-in Information

Work sheets and internal chain-of-custody are sent to the various departments (i.e. inorganics, GC/MS, GC, etc.)

The SC or a designee will log in samples. The date on the receipt form will be the date of sample receipt in the laboratory. The time, date of collection, and recipient name will be noted in the remarks column of a COC sample log-in sheet (Figure 3.8); and the original receipt documentation will be attached to the COC record.

Sample Log-in Document Storage

There will be two repositories for documents associated with a project. The first repository is the project file. This file will contain the following documents:

- Contracts, purchase orders, task order, and/or other work authorization
- Original project sheet
- Computer-generated project sheet
- Project modification forms

The second repository for related documentation is the analytical data file, which will contain a copy of the final project report/QC report and any other documents related to the project analysis (i.e. 1) signed airbill; 2) signed chain-of-custodies; 3) work sheet; 4) sample tags; 5) traffic reports; 6) bench sheets; 7) raw data).

Corrections to Documentation

When it becomes necessary to make corrections to any form of documentation (e.g., sample tags, COC forms, daily logbooks), the obsolete information is crossed out with a single line and the changes are made and initialed by the person making the change.

3.9 SAMPLE PACKAGING AND SHIPPING

Preparation of Samples for Shipment

The following is a description of the procedure followed when transporting environmental samples from the sampling site to the laboratory:

- The outer surface of all sample containers is cleaned with bottled water and paper towels.
- Sample collection points, depth increments, and sampling devices are identified and documented.
- Log book entries, sample tags, COC forms, and field record sheets with sample identification points, date, time and names or initials of all persons handling the sample in the field are completed.
- Custody tape is wrapped around the neck and cap of each container.
- Samples and trip blanks are placed into a sample cooler provided by the laboratory along with blue ice packs. After a cooler is filled, the appropriate COC form is placed inside the cooler

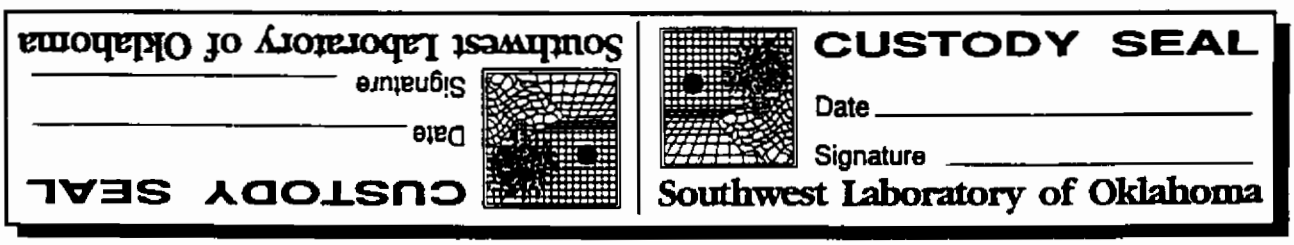


FIG. 3.15 Example Custody Seal for Sample Containers/Coolers

and the outer surface of the cooler is cleaned.

- Glass sample containers are wrapped with plastic insulating material to prevent contact with other sample containers or the inner walls of the cooler.

- Once all packaging is completed, each cooler is sealed with identifying labels/custody seals (see Figure 3.10) which are initialed by the field sampler for COC procedures. Custody seals are placed across the binding tape that secures the lid of the shipping container for both the front and back side of the container. For the back side, the custody seal is placed across the hinge, if possible.

- Samples are classified according to the Department of Transportation (DOT) regulations pursuant to Title 49 CFR.

- The laboratory is then notified prior to shipment that samples are being sent to the laboratory for analysis. This notice should be given at least 24 hours in advance of the expected sample arrival date. Notification includes shipping information (i.e., airbill number, courier company, number of shipment containers to be sent).

Shipping Containers

Samples are packaged in thermally insulated, rigid coolers, according to DOT specifications 173.510 and 172 Subparts B, C, and D, and Subparts A and B of Part 173. Sample containers are placed in a cooler that contains blue ice and absorbent packing for liquids or styrofoam packing for solids. The completed COC form is placed inside the shipping container, unless otherwise noted.

Marking and Labeling

The cooler is marked as follows:

- Proper shipping name: Hazardous substance, liquid, or solid

- Hazardous class: To be determined (label placed in upper left corner of outer container)

- Labels: "This Side Up" or arrows placed on the opposite side of the outer container if a liquid is to be shipped
- Custody tape is wrapped twice, in a single strip, around the outside of each cooler and initialed.

A hazardous material shippers certification is filled out and will accompany the shipment. The container is secured with strapping tape to prevent leakage.

Shipping Transportation/ Courier

It is recommended that an overnight express service (i.e., Federal Express) be used for sample transport. If an air freight service is used, samples can be picked up at the airport (located 20 minutes from the laboratory). When samples are scheduled to arrive at the laboratory on a Saturday, the laboratory must receive notice of this shipment at least 24 hours in advance.

4.0 CALIBRATION PROCEDURES AND FREQUENCIES

Calibration is the process for determining the correctness relative to physical or chemical standards used or assigned values in scales of measuring instruments. It establishes a reproducible reference point to which all sample measurements can be correlated. Instruments are calibrated as per method (i.e., SW846 CLP-SOW, etc.) The following are examples of those calibrations.

4.1 FIELD EQUIPMENT

All field equipment is calibrated daily prior to use, and immediately recalibrated if field personnel suspect that the calibration may have been altered. Reasons for such alteration include change of batteries, equipment being dropped or knocked around, or significant changes in temperature since the last calibration. Since instrumentation and procedures are continually being updated, field personnel are required to consult the appropriate instruction manual for calibration instructions.

Specific Conductance Meter

- With the instrument turned off, check the meter's mechanical zero setting. Adjust the screwdriver adjustment control on the meter face if necessary to obtain a zero reading.

- Press the power switch to on and press the battery check switch. Verify the meter needle deflects to the battery check area.

- Connect a clean, dry probe to the instrument. Remove the instrument from its carrying case and place it on a padded surface.

- Press the 0-2 range switch and verify that the meter reads zero. If it does not, adjust the null adjust potentiometer RB9 on the amplifier circuit board to obtain a reading as near zero as possible.
- Press the 0-2000 range switch.

- Immerse the probe in the 1000 mg/l sodium chloride solution. The meter should read 1990 uhos/cm. If it does not, adjust the standardization potentiometer R32.

pH Meter

- Connect the two probes to the appropriate jacks on the instrument panel. Be sure that fill hole in the pH electrode is uncovered. The fill hole is to remain covered at all times except for calibration and pH measurement. When the electrode is not in use, slide the rubber sleeve over the fill hole.

- Prepare pH 4 and pH 9 buffer solutions by dissolving the contents of one powder pillow of each in separate beakers containing 50 mL demineralized water.

- Select the T(c) mode and measure the temperature of each buffer solution. Referring to the temperature coefficients table, determine the actual pH values of the buffer solutions for those temperatures. Calculate the difference between the two pH values.

- Immerse the probes in the pH 4 buffer solution and select the pH mode. Allow approximately 30 seconds to reach equilibrium and adjust the CAL control to obtain 0.00 reading.

- Remove the probes from the pH 4 solution and rinse thoroughly with demineralized water.

- Immerse the probes in the pH 9 buffer solution. Allow approximately 30 seconds for the probes to reach equilibrium. Adjust the SPAN control for a reading equal to the difference value calculated above.

- Adjust the CAL control for a reading equal to the actual pH value of the pH 9 buffer as adjusted for temperature.

- Rinse the probes thoroughly with demineralized water.

Water Level Indicator

This instrument arrives calibrated by the manufacturer for water level measurement.

Digital Thermometer

This instrument is calibrated against an NBS thermometer at least 2 points within the anticipated range.

Geiger Counter

Geiger counters are sent annually to an independent contractor for calibration.

4.2 LABORATORY INSTRUMENTATION —INORGANIC CHEMISTRY SECTION

Atomic Absorption Spectrophotometer Systems (AAS)/Inductively Coupled Argon-Plasma Emission Spectrophotometer (ICAP)

For AAS systems (i.e., flame AA, graphite furnace AA and ICP), the instruments are calibrated daily, and each time the instrument is set-up. Appendix C lists the instrument operating parameters employed in the analysis.

Calibration standards are prepared fresh before each analysis and are discarded after use. These calibration standards are checked for traceability using NBS reference standards or EPA QC solutions. Appendix D contains a list of NBS and EPA reference standards available for laboratory use.

The following QA/QC requirements are employed for the AA calibration:

a. Initial Calibration Verification (ICV)

- The accuracy of the initial instrument calibration must be verified and documented for every analyte by the analysis of an Initial Calibration Verification solution (ICVS - i.e., an NBS, SRM or EPA QC solution). When measurements exceed the control limits of the ICVS or within the target range values supplied by the QC solution, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

- If the ICVS is not available, or if a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for calibration, but within the calibration range. (See Appendix D for Criteria and Guidelines for Standard Traceability)

- The ICVS must be run at each wavelength used for analysis.

b. Calibration Blank

- Must be analyzed each time the instrument is calibrated.

- Must be analyzed at the beginning and the end of the run, and at a frequency of 10% during the run. It is also analyzed after a standard run or after a contaminated sample run to check for carry-over contamination.

- Blank results are to be reported down to the instrument detection limits (IDL).

- If the result is greater than method detection limit (MDL), the analysis is terminated, problem corrected, the instrument recalibrated and calibration reverified.

c. Continuing Calibration Verification (CCV)

- CCV must be performed for each analyte at a frequency of 10% or every two hours during analysis, whichever is more frequent.

- CCV must be also analyzed for each analyte at the beginning and at the end of the analysis.

- The analyte concentration in the CCV must be near the mid-range of the calibration curve.
- The same calibration standard must be used throughout the analysis for a particular case.

TABLE 4.1 INTERFERANT AND ANALYTE ELEMENTAL CON-CENTRATIONS USED FOR ICP INTERFERENCE CHECK SAMPLE

Analytes	(mg/l)	Interferants	(mg/l)
Ag	1.0	Al	500
Ba	0.5	Ca	500
Be	0.5	Fe	200
Cd	1.0	Mg	500
Co	0.5		
Cr	0.5		
Cu	0.5		
Mn	0.5		
Ni	1.0		
Pb	1.0		
V	0.5		
Zn	1.0		

■ One of the following standards can be used for continuing calibration verification:

1. EPA Solution
2. NBS SRM
3. In-house prepared solution from an independent standard

■ If the CCV results exceed the specified control limits (i.e., 95% confidence limits of the true values or the given target range values), the instrument must be recalibrated and the preceding 10 samples or less reanalyzed for the analytes affected.

d. ICP Interference Check Sample Analysis (ICS)

■ To determine if interelement and background correction is required, ICS is analyzed at the beginning and end of each sample analysis run (minimum of 2 times for every 8 hours).

■ Results must fall within the control limits of ± 20% of the EPA supplied true values for the analytes included in the ICS. (See Table 4.1 for list of analytes and interferants in the ICP check sample). If not within the control limits, the analysis is terminated, the problem is corrected, the instrument is recalibrated, and the samples are reanalyzed.

■ If an EPA ICP check sample is not available, an independent ICP check sample is prepared with the interferant and analyte concentration at the levels specified in Table 4.1. The mean value and the standard deviation is established by initially analyzing the prepared check sample at least 5 times for each parameter. Control limits are then established for the in-house prepared solution. It must fall within ± 20% of the mean value.

If an interference cannot be resolved successfully, a standard addition technique will be used for both AA and ICAP. Standards of the analytes will be added to the duplicate sample and the concentration of the analyte(s) can be determined by difference.

The following requirements are employed when using a method of standard addition for graphite furnace analysis:

■ Data must be within the linear range determined by the calibration curve.

■ The sample and the three spikes must be analyzed consecutively.

■ Only single injections are required.

- Spikes should be prepared such that:

  1. Spike 1 is approximately 50% of the sample absorbance.

  2. Spike 2 is approximately 100% of the sample absorbance.

  3. Spike 3 is approximately 150% of the sample absorbance.

Spectrophotometers

The manufacturer instructions for instrument operation are followed for proper operating procedures.

Spectrophotometers are calibrated daily prior to any sample analysis. The calibration standards are prepared from reference materials or commercial standards (traceable to EPA or NBS reference materials) at a minimum of three concentrations, including a calibration blank to cover the anticipated range of measurements. The requirement for an acceptable initial calibration is a correlation coefficient equal to or greater than 0.996 (based on statistical historical data). Before sample analysis, an initial calibration verification standard is analyzed. The response of this standard must be within 95% confidence limit of the true values or target range values provided by the QC sample. If not, the instrument must be recalibrated.

The instruments are also checked for wavelength calibration. For UV/IR instruments, polystyrene film is used to calibrate the wavelength.

All absorption cells (i.e., cuvettes, quartz cells) are kept clean, free of scratches and fingerprints, and are rinsed with the solution to be analyzed prior to use. Matched cells are checked to see that they are equivalent by placing portions of the same solution in both cells and taking several readings of the transmittance or absorbance.

Total Organic Carbon Analyzer (TOC)

For TOC calibration, a known volume of potassium hydrogen phthalates (KHP) solution is analyzed as the calibration solution on the carbon analyzer. A minimum of three calibration solutions encompassing the linear range of the carbon analyzer is prepared and analyzed. Linear regression analysis of standard concentration in ug C versus response in millivolts (mv) is done to obtain a calibration curve. The linear regression fit must be equal or greater than 0.99 (based on statistical evaluation of historical calibration data) or the standards are rerun and a new regression is calculated.

4.3 LABORATORY INSTRUMENTATION —ORGANIC PROCESSING SECTION

Gel Permeation Chromatograph (GPC)

The following procedure is employed for the calibration of the GPC system:

- Packing the column - Place 70 grams (g) of Bio Beads SX-3 in a 400 milliliter (mL) beaker. Cover the beads with 50/50 methylene chloride and allow the beads to swell overnight before packing the column. Transfer the swelled beads to the column and start pumping solvent through the column, from bottom to top, at 5.0 mL/minute. After approximately 1 hour, adjust the pressure on the column to 7-10 psi and pump an additional 4 hours to remove air from the column. Adjust the column pressure periodically as required to maintain 7-10 psi.

- Prepare the GPC calibration solutions as follows: (1) Corn oil - Add 25 mg corn oil to sufficient amount of methylene chloride to attain a final volume of 250 for a 100 mg/mL; (2) phthalate-phenol - Add 1.0 g of Bis(2-ethyl hexyl) phthalate to sufficient amount of methylene chloride for a final volume of 250 ml and a final concentration of 4 mg/ml. Add 0.15 g of neat pentachlorophenol into approximately 250 ml methylene chloride for a final concentration of 0.6 mg/ml.

■ Calibration of the column - Load 5 mL of the corn oil solution into sample loop No. 1 and 5 mL of the phthalate-phenol solution into loop No. 2. Inject the corn oil and collect 10 mL fraction (i.e., change fraction at 2-minute intervals) for 36 minutes. Inject the phthalate-phenol solution and collect 15 mL fraction for 60 minutes. Determine the corn oil elution pattern by evaporation of each fraction to dryness followed by a gravimetric determination of the residue.

■ Analyze the phthalate-phenol fractions by GC/FID on the DB-5 capillary column.

■ Plot the concentration of each component in each fraction versus total eluent volume (or time) from the injection points. Choose a "dump time" which allows $\geq$85% removal of the corn oil and $\geq$ 85% recovery of the bis(2-ethylhexyl)-phthalate.

■ Choose the "collect time" to extend at least 10 minutes after the elution of pentachlorophenol. Wash the column at least 15 minutes between samples.

■ Typical parameters selected are: Dump time = 21 minutes; collect time = 24 minutes; and wash time = 15 minutes; Elute volume collected = 120 ml.

General Laboratory Equipment

Balances are calibrated before every use with standard Class-S calibration weights and are calibrated annually by a licensed specialist. The pH/specific-ion meters are calibrated before each use with a minimum of three standard solutions. (See Appendix D, Standard Operating Procedures for pH meter, balances, and conductivity meter for discussion)

4.4 LABORATORY INSTRUMENTATION —CHROMATOGRAPHY SECTION

Gas Chromatographs (GC)

Injection of secondary standards, validated by the use of EPA or NBS reference standards, is used to adjust the sensitivity and selectivity of the analytical system for each compound being analyzed. The system is calibrated by preparing standards at a minimum of five concentration levels for each analyte. The low-level standard is at or near the established detection limit. The medium- and high-level standards are at concentrations that correspond to the expected range of concentrations found in the samples. These standards will define the working range of the GC detector. (See Appendix D, Traceability and List of EPA and NBS reference standards available)

The results of standard calibrations (low, medium, and high ranges) for each analyte are tabulated with respect to response versus concentration. The ratio between response and concentration, known as the response factor (RF), can be used to prepare a calibration curve for each compound. It is expressed in an equation as:

$$RF = \frac{\text{Area response of analyte}}{\text{Concentration of analyte in the standard}}$$

The following criteria are employed for the GC linearity calibration:

a. Initial Calibration Verification

■ % Relative Standard Deviation (RSD) cut-off for RF = $\pm$ 20% for all analytes except for problematic (i.e., poor responder) analytes (e.g., gases, endrin, DDT, DDE, DDE, DDD, etc.). RSD is calculated as:

$$\%\,RSD = \frac{s \times 100}{\text{average RF of the individual analytes in the standard solution}}$$

where s = standard deviation of the RF of each analyte

and where Average RF = mean of the RF of the analyte

■ %RSD cut-off for problematic compounds = ± 50%

■ If an analyte in the % RSD determination of the linearity standard check is greater than 20% , or 50% for problematic compounds then linear regression or a straight-line curve is used to determine linearity. (Alternatively, there is an option to use the quadratic equation or a point-to-point curve if the correlation coefficient for the linear regression is less than 0.995.)

■ If the compounds are still out of criteria after using linear regression, the quadratic equation, or a point-to-point curve, reanalyze the standard concentration that is of suspect and then recalculate % RSD.

■ If the compounds are still out of criteria after such reanalysis of the suspected standard, corrective and/or preventive maintenance should be performed to check the system. A new set of initial calibration curves is to be analyzed.

b. Continuing Calibration Verification

■ Percent Difference (% D) cut-off for RF = ± 15% for all analytes except for problematic (i.e., poor responder) analytes (e.g., gases, endrin, methoxychlor, endrin aldehyde). % D is calculated as:

$$\%\,D = \frac{[\text{average RF - RF (continuing calibrationstandard)}] \times 100}{\text{mean of average RF}}$$

■ % D cut-off problematic compounds = ± 50%

■ If 20% of the analytes in the % D determination of the standards for linearity check have values greater than 35%, then linear regression or a straight-line curve is used to determine linearity. (Alternatively, there is an option to use the quadratic equation or a point-to-point curve if the correlation coefficient for the linear regression is less than 0.995.)

■ If the compounds are still out of criteria after using linear regression, the quadratic equation, or a point-to-point curve, reanalyze the continuing calibration standard concentration and recalculate % D.

■ If the compounds are still out of criteria after such reanalysis, corrective and/or preventive maintenance should be performed to check the system.

■ A new set of initial calibration curves is to be analyzed.

c. The continuing calibration standard is analyzed every 12 hours during sample analysis.

d. A solvent blank using solvent (Pesticide grade) suitable for the detector is used to check system contamination and is also analyzed after a standard run or after a contaminated sample has been analyzed to check for carry-over contamination.

NOTE: Cut-off criteria for initial calibration and continuing calibration are interim guidelines. The GC laboratory will set new criteria based on historical data obtained from previous calibration standards used for the various analytical methods.

High Performance Liquid Chromatographs (HPLC)

The system is calibrated by preparing standards at a minimum of five concentration levels for each analyte. The low-level standard is at or near the established detection limit. The medium- and high-level standards are at concentrations that correspond to the expected range of concentrations found in the samples. These standards will define the working range of the HPLC. Continuing calibration is analyzed after every 10 samples and at the end of run. Criteria for % RSD for the initial calibration is within 20% or a linear regression or a straight-line curve is used to determine linearity. If the correlation coefficient is less than 0.995, corrective and/or preventive maintenance is done, and a new set of calibration curves is analyzed. Criteria for % D for continuing calibration is within 15% or a new standard is analyzed and % D recalculated.

Ion Chromatographs (IC)

The calibration procedure for the system is the same as the HPLC.

4.5 LABORATORY INSTRUMENTATION—MASS SPECTROMETRY SECTION

Procedures for calibration and instrument tuning for sensitivity and selectivity are somewhat similar to those for gas chromatography method. The primary difference between GC and GC/MS methods is concerned with the validation of the mass spectrometer as the detector. GC detectors generally operate by sensing a change in an electrical field (e.g., GC-EC, GC-FID, Hall); whereas, mass spectrometers sense a change in charge with reference to the mass of the compound. Further, the charge molecule ion will fragment reproducibly into an array of ions. The result is a characteristic mass spectrum of the compound. The first step in the calibration of the GC/MS system is to demonstrate the ionization and fragmentation of standard mass spectral tuning compounds. This is accomplished, as well as a sensitivity check, with the use of two EPA-specified compounds injected. Those compounds are: 4-Bromofluorobenzene (BFB) for volatiles and Decafluorotriphenylphosphine (DFTPP) for semivolatiles. These standards are run daily to validate the GC/MS system tune. (See Tables 4.2 and 4.3, for Tune Criteria)

Calibration of the GC/MS, like that of GC calibration, is established and validated by the injection of EPA traceable standards at a minimum of five concentration levels over the range of likely sample concentrations. An internal calibration procedure is used: in addition to surrogate recovery compounds, sample extracts are spiked with internal calibration standards that span the retention time range of the analytes of interest. The concentration of the analytes is calculated with reference to the RF of the internal standards for each sample. RF is defined as:

$$RF = \frac{(A_x)\ (C_{is})}{(A_{is})\ (C_x)}$$

where:

A_x = area of the characteristic ion for the measured compound

A_{is} = area of the characteristic ion for the specific internal standard

C_{is} = concentration of the internal standard (ng/μL)

TABLE 4.2 BFB KEY IONS AND ABUNDANCE CRITERIA

MASS	ION ABUNDANCE CRITERIA
50	15.0 - 40.0 percent of the base peak
75	30.0 - 60.0 percent of the base peak
95	base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of base peak
173	less than 2.0 percent of mass 174
174	greater than 50.0 percent of the base peak
175	5.0 - 9.0 percent of mass 174
176	greater than 95.0 percent but less than 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

TABLE 4.3 DFTPP KEY IONS AND ABUNDANCE CRITERIA

MASS	ION ABUNDANCE CRITERIA
51	30.0 - 60.0 percent of mass 198
68	less than 2.0 percent of mass 69
70	less than 2.0 percent of mass 69
127	40.0 - 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent relative abundance
199	5.0 - 9.0 percent of mass 198
275	10.0 - 30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0 - 23.0 percent of mass 442

C_x = concentration of the measured compound (ng/μL)

Further precision, accuracy, and continuing calibration are demonstrated with the use of repeated analyses of spiked and duplicate spiked samples and EPA check samples. Reagent blanks are analyzed in each batch of semivolatile analyses and are analyzed daily for volatile organic analyses.

The QA/QC requirements employed for the GC/MS calibration are discussed in the following subsections.

Instrument Tuning

- EPA-CLP (2/88 SOW) tune criteria for BFB and DFTPP are used before any sample analysis for VOA and BNA. (See Tables 4.2 and 4.3, BFB and DFTPP Tune Criteria)

- Other tuning criteria can be used for CLP 3/90 SOW or the Superfund Method for Low Concentration Water.

- A tune criteria is required every 12 hours during sample analysis.

Initial Instrument Calibration

- For EPA-CLP (2/88 SOW) and EPA SW846 Method 8240 and 8270 the criteria for VOA and BNA calibration check compounds (CCC) and system performance check compounds (SPCC) is employed.

- BNA and VOA CCC are:

BNA CCC	VOA CCC
Phenol	Vinyl Chloride
1,4-Dichlorobenzene	Chloroform
2-nitrophenol	1,2-Dichloropropane
Hexachlorobutadiene	Toluene
4-Chloro-3-Methylphenol	Ethylbenzene
2,4,6-Trichlorophenol	
Acenaphthene	
N-Nitrosodiphenylamine	
Pentachlorophenol	
Fluoranthene	
Di-n-octyl phthalate	
Benzo(a)pyrene	

% RSD is calculated for all compounds. It should be within ± 30% for all CCC compounds and should be within ± 30% for all other compounds.

- BNA and VOA SPCC are:

BNA SPCC	VOA SPCC
N-Nitroso-di-n-Propylamine	Chloromethane
Hexachlorocyclopentadiene	1,1-Dichloroethane
2,4-Dinitrophenol	Bromoform
4-Nitrophenol	1,1,2,2-Tetrachloroethane
	Chlorobenzene

The average RF is calculated for all compounds. RF for both BNA and VOA SPCC compounds must be at least 0.300 except for bromoform which must be at least 0.250. The RF for all other BNA and VOA compounds must be at least 0.05.

For EPA-CLP 3/90 SOW all compounds must meet a minimum Relative Response Factor (RRF). most compounds must meet a maximum %RSD (20.5%) criteria.

Continuing Calibration

- Same list of CCC and SPCC for BNA and VOA analysis.

- CCC and SPCC criteria

  1. % D must be less than 25% for all CCC and should be less than 25% for all other compounds for BNA and VOA analysis.

  2. The RF is calculated for all compounds. RF for all other BNA and VOA SPCC compounds must be at least 0.300, except for bromoform which must be at least 0.250. The RF for all other BNA and VOA compounds must be at least 0.05.

- Continuing calibration must be performed after tune criteria is met.

- Continuing calibration must be performed before beginning sample analysis and must be done every 12 hours during analysis for BNA and VOA.

■ For EPA-CLP 3/90 SOW all compounds must meet a minimum RRF. Most compounds must meet a maximum %Difference (25%) criteria.

Internal Standard

Internal standard areas are monitored as a measure of the GC/MS instrument calibration. The areas of each internal standard in each sample are compared to the internal standard areas in the continuing calibration standard associated with the samples. If the samples are analyzed under the same tune as the initial or continuing calibration, the areas in each sample are compared to those in the VOA or BNA continuing calibration standard in the initial or continuing calibration.

■ The area and retention time for each internal standard from the calibration standard and the upper and lower limits of the EICP area should be within -50% to +100%.

■ When the retention time of any internal standard changes by more than 30 seconds, the system must be inspected and corrections made.

■ The EICP area of each internal standard must fall within the limits of the 12 hour standard.

5.0 ANALYTICAL PROCEDURES

5.1 FIELD TESTING AND SCREENING

During multimedia sampling activities, selected physical and chemical parameters in the air, water, and soil at the site are measured. Equipment and general procedures for analysis of field samples are listed below. Because field instrumentation and analytical methodology is continually being updated, field personnel are required to consult each manufacturer's instruction manual for operating procedures.

Measurements of pH

Measurements of pH are taken on water purged from wells prior to sampling. Groundwater samples are collected after a stable pH is achieved to ensure that conditions are representative of the formation. Measurements for pH also are taken on surface water samples. A Orion Model 231 Digital pH meter or equivalent is used for field analyses using the procedure described below.

Rinse the probes thoroughly with ASTM Type II reagent water to prevent any carryover. Make certain that the pH electrode fill hole is uncovered and the mode switch is set to pH. Immerse the probes in the test sample and take the reading. Allow 30 seconds for the reading to stabilize. After pH measurements are conducted, slide the rubber sleeve over the fill hole.

Specific Conductance

Conductivity measurements are taken on purge water and all groundwater and surface water samples using a Hach Model 16300 portable conductivity meter according to the procedure described below.

Connect the probe to the PROBE INPUT connector on the front panel. Press the POWER switch on and perform a battery check. Press the switch for the highest range and immerse the probe in a beaker containing the sample solution. Shake or tap the probe on the bottom of the beaker to ensure that no air bubbles are trapped near the electrode. Allow about 10 seconds for the probe to stabilize before taking the reading. If the meter indicator falls in the lowest 10 percent of the range, switch to the next lower range. Repeat until the proper range is selected. Dilute if necessary. Rinse the probe with ASTM Type II water between each use.

Temperature

Temperature data is used in conjunction with the chemical data for groundwater and surface water characterization. Temperature measurements are taken on purge water and all groundwater and surface water samples using a mercury thermometer or the conductivity meter, which is equipped to measure temperature, according to the procedure described below.

Connect the temperature probe and select the T(c) mode. Immerse the probe in the test sample and allow 30 seconds for equilibration. Take the reading. Rinse the probe with ASTM Type II reagent water after each use.

5.2 LABORATORY METHODS

References

The laboratory follows analytical procedures based on EPA-approved methods for both inorganic and organic analyses of multimedia environmental samples. Methods used for inorganic and organic analysis of routine samples are based on 40 CFR part 136 as published in the Federal Register (October 26, 1984), the EPA's Test Methods for Evaluating Waste (SW-846, 3rd edition), and Standard Methods for the Examination of Water and Wastewater, 16th Edition 1985. Unless otherwise notified by the client, the above methods shall be followed. Appendix A, provides a listing of analytical protocols utilized by the laboratory. A listing of our most utilized methods is found in Appendix B & Appendix E.

If typical methods are rendered ineffective by matrix interferences or if analytical parameters of detection limits, precision, specificity, etc., would require method variance (i.e., modification of the method), the Project Officer will notify the client/contractor of the method modification. A copy of the variance will be sent to the client/contractor to seek approval of the method change. The modification request must show that the conditions for the laboratory variance are similar to the expected conditions (i.e., sampling and handling techniques, environmental matrix concentration range, interferences, etc.) in the EPA approved methods.

Changes in operations prior to instrumental analysis (e.g., sample preparation and storage) must be documented.

Documentation

The objective of document control is to assure that all documents for a given program are accountable and traceable. It includes chain-of-custody records, all logbooks, graphs, and other miscellaneous items.

Record-Keeping

Documentation in the laboratory is initiated by the SC who receives samples, assigns laboratory numbers and generates COC forms which document sample movement in the laboratory. Each shipment of samples received is given a unique batch number (project number). A batch consists of a number of samples carried through the entire analytical procedure, along with samples and standards. All work performed on a sample batch is documented in a bound laboratory logbook which is described as follows:

1. Sample Receiving Logbook: It is a compilation of computer-generated sample summary forms which were entered into the laboratory sample data base on a sample receipt basis. It is compiled on a monthly basis to document sample receipt information.

2. Instrument Maintenance Logbook: To record the maintenance performed on the analytical instruments. It is maintained for each GC, GC/MS, AA, or other analytical instrument.

3. Standards Logbook: To record the preparation and use of all standards in the laboratory. It shall indicate standard traceability to EPA or NBS standards. It shall note date of preparation, concentration and by whom, as well as date of expiration of the stock standards or reagents.

4. Chemist's Notebook: To record the raw data and final data of every batch. It is used to document all activities associated with the analytical process. Laboratory notebook of each staff is a functional record and is pre-numbered.

5. Instrument Benchsheet Logbook: To record sample run sequence or injections done in a day's or shift's run.

Rules Governing the Use of Logbooks

a. Bound logbooks with pre-numbered pages are the preferred record-keeping forms. Loose sheets are not to be used unless permanently affixed to the logbooks.

b. Only assigned laboratory notebooks or logbooks are used for record-keeping related to project work.

c. All writing must be legible and shall be completed in ink. All numbers must be clear. Corrections should be made by drawing one line through the incorrect entry, entering the correct information, and initialing the change.

d. Complete information should be entered so that in an examination it can be determined what was done, when, and what the results were.

e. If any data are determined to be invalid, reasons are indicated.

f. All relevant information is included (e.g., the manufacturer and lot number of a chemical, the specific procedure used for sample preparation and analysis, instrumental conditions, etc.).

INORGANICS DOCUMENTS
- File inventory
- Chain-of-custody record(s)
- Sample tag(s)
- Airbill(s)
- Inorganics Traffic Report(s)
- Inorganics Analysis Data Summaries
- Copies of analysts' notebook pages and/or benchsheets
- Worksheets
- ICAP and AA instrument logbook pages
- Sample tracking documents
  - Sample receipt log pages
  - Internal custody records
- Copies of instrument printouts
- QA/QC data reports
- Duplicate, blank, etc., analyses results
- Related correspondence and memos
- All other related documents

FIG. 5.1 Inorganics Document Inventory Check List

g. When work is continued in another notebook or logbook, the number of the first notebook is written in the first page of the second notebook and vice-versa.

Document Control

Document control is accomplished through the use of a centralized repository document inventory and a Data Clerk with purview over all the documents generated in conjunction with the project or contract. All project files and analytical data files and related documentation to sample analysis are maintained by the Data Clerk.

Document Handling

The Data Clerk is responsible for the collection, organization, maintenance and security of all documents. The Data Clerk will establish a client/contract file for all documentation regarding a project or a contract.

A client/contract file is generated when the project file (i.e., sample receipt and log-in documents) are transferred by the SC to the Data Clerk for custody. Within a project file, subfiles are established for each major element of the contract. These files are stored in a locked file cabinet in the Data Clerk's office.

Each file contains the following type of documents:

a. Project File: All documentation relating to sample receipt and log-in. This also contains project sheet information as well as contract information, etc.

b. Analytical File: This contains the final data/QC report and raw data relating to sample analysis. The order of filing within the analytical file would consist of final data/QA report, raw data (i.e., chromatograms, RIC/Quan, strip charts, etc., or any instrument's data recording output).

c. Miscellaneous File: This will contain the instrument benchsheets, all documents associated with sample preparation (i.e., analysis request form), analyst's notes, etc. In the case of laboratory notebooks and instrument benchsheets, photocopies will be used in place of the originals being maintained by the analyst.

Consistency of Documentation

Before releasing analytical results, the laboratory assembles and cross-checks the information on sample tags, custody records, laboratory benchsheets, personal and instrument logs and other relevant data to ensure that data pertaining to each particular sample or case is consistent throughout the record. (See Data Collection, Validation and Reporting Section for details)

Document Inventory

Document tracking and control are facilitated through the use of an inventory checklist for document tracking. Figures 5.1 and 5.2 show the checklist for both inorganic and organic parameters.

DOCUMENT INVENTORY LIST - FORMAT

SECTION I
Narrative

SECTION II
QC Summary

SECTION III
Sample Data
Traffic Reports
One-entry for each Sample

SECTION IV
Lab Detection Limits
VOA Initial Calibration Data Form VI
VOA Continuing Calibration Data Form VII
BNA Initial Calibration Data Form VI
BNA Continuing Calibration Data Form VII
Pesticide Data Summary Forms VIII-X
VOA Initial Calibration Raw Data
VOA Continuing Calibration Raw Data
BNA Initial Calibration Raw Data
BNA Continuing Calibration Raw Data
Pesticide Raw Data

SECTION V
DFTPP Raw Calibration Data
BFB Raw Calibration Data
Method Blank Data
Matrix Spike Data
Matrix Spike Duplicate Data

SECTION VI
Airbill/Shipping Manifest
Field Chain-of-Custody Document
Sample Receipt Form

SECTION VII
Lab Notebook Pages
GC Injection Log Pages
GC/MS Injection Log Pages
Miscellaneous File
Pesticide Raw Data File

FIG. 5.2 Organics Document Inventory List

Handling Confidential Documents

All documents received with a group of samples and/or generated in the course of analysis shall be kept confidential. Documents specifically marked confidential that may accompany the samples, are to be treated separately from other case-related documents.

The following procedures are employed for handling documents marked CONFIDENTIAL:

1. Client/contractor is contacted to assure that receipt of these documents is correct and required for analysis, and returned as directed by client/contractor.

2. If the documents are necessary to begin the sample analyses, the documents are placed in a secured file separate from the regular files and under the control of the Data Clerk. The Data Clerk keeps these documents secured at all times and only authorized personnel have access to them.

Document/Data Package Shipping.

The delivery schedule of the data package depends on the contract requirements. The date of shipping is documented and a list of data/documents shipped is retained for record. A copy of the data package sent is kept by the laboratory to be filed for future reference in case of client's future request for information.

Standard Operating Procedures

The laboratory maintains SOPs for each laboratory section that describe standard procedures used by each laboratory section for use of logbook,

benchsheets, traceability of standards, instrumentation, sample and environmental data.

The laboratory maintains Standard Operating Procedures (SOPs) for every major analytical procedure. These procedures detail use of logbooks, benchsheets, traceability of standards, instrumentation, sample, and environmental data. SOPs are updated yearly with any changes from previous revisions approved by the Section Manager/Supervisor and/or QC Officer. All SOPs within the laboratory are the current revision which are dated and controlled by the use of a sign-off sheet documenting who has the procedure and which revision they have. Out of date material is "retired" by removal from circulation and maintained in a locked file and used for reference material only.

Document Control, Review, & Approval

Documents are updated for any revision made. Changes made in documentation shall reflect the actual procedures being followed. Before any revision is made, analyst concerned for revision of such documents shall submit to the Section Supervisor or the Project Officer (PO) the proposed revisions.

If the revision is justified for the changes to be done, the Section Supervisor or the PO submits the proposed revision of the standard operating procedure (SOP) to the QA Officer for approval.

Document revision shall also include policy changes which could substantially impact the QA/QC plan. They are as follows:

- Personnel changes relating to QA/QC responsibilities
- Method changes

All Document revisions are reviewed by two supervisors (i.e., managers, QA Officer, etc.). Approval signatures are on the cover with revision numbers and date of revision. Each copy is given a document control number.

Outdated documents are collected, and the latest revision issued. The outdated document is "retired," and entered into the retired document log. Copies are maintained for historical purposes.

Standards Preparation

All inorganic and organic analytical standards utilized for instrument/methodological calibration and preparation of QC samples are traceable to SRMs. Primary standards are obtained from reliable, certifiable source, and of highest possible purity. Prepared commercial standards are verified against SRMs from EPA or NBS for traceability. Appendix IV contains Standard Operating Procedures for Standard Traceability, Expiration, and Criteria and Guidelines used by the laboratory for standard preparation.

5.3 TYPES OF DETECTION LIMITS

Instrumental Detection Limit (IDL)

It is defined as the smallest signal above background noise that an instrument can detect reliably. It does not address possible blank contaminants or matrix interferences.

IDL for each analyte in a given method is determined for each analytical instrument used. It is quarterly updated to verify instrument sensitivity changes. The following procedure is used to determine IDL:

- Using EPA or NBS supplied SRMs, if available, perform seven consecutive measurements of standards for all components being measured at 3 - 5 times the required detection limit concentrations [i.e. MDL or contract required detection limits (CRQL) on three nonconsecutive days.
- These analyses are performed using the instrumental working conditions specified in the method, on standards in appropriate solvent for base/neutrals, acids, and pesticides/PCBs; standards diluted into reagent water for volatile organics; and trace metal analytes in reagent water.
- IDL is determined by multiplying 3 times the average of the standard deviations of the measured values.

Limit of Detection (LOD)

LOD is the lowest concentration level of an analyte that the analytical process can reliably detect. Sometimes, the IDL and LOD are operationally the same since an indication of whether an analyte signal exceeds peak-to-peak noise. LOD accounts for blank contamination but not for matrix complexity and interferences. It is also numerically equivalent to the MDL as the value for blank approaches zero. The recommended value for LOD is 3s where s is the standard deviation of the difference between total value measured for the sample and value measured for the blank. It is expressed in an equation as:

$$S_T - S_B > 3s$$

where

S_T = Total value of the analyte measured in the sample

S_B = Value of the analyte measured in the blank

s = Standard deviation for these measurements ($S_T - S_B$)

Method Detection Limit (MDL)

MDL is defined as the minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero. It also refers to the minimum concentration of an analyte that a method can detect reliably in either a given sample matrix or blank.

It is expressed in an equation as:

$$MDL = \frac{Ks}{M}$$

where:

K = 3

s = Standard deviation of average noise level

m = Slope of the calibration curve

Practical Quantitation Limits (PQL)

PQL is the lowest level that can be reliably achieved within the laboratory control limits of precision and accuracy during routine laboratory operating conditions. It is expressed in an equation as

PQL = [MDL x factor]

For nonaqueous samples, the factor is on a wet-weight basis. (See Table 5.1)

Methods for Which Limits of Detection Are to Be Developed

The laboratory periodically reevaluates the MDLs for the analytical methods used.

MDL established by the laboratory for EPA approved methods (i.e. Federal Register 600 series, SW-846 "7000" series, and Methods of Chemical Analysis of Water and Wastes) shall be compared against the MDL defined in these methods to determine if in-house quality control procedures are effective. In instances where an EPA method is used in analysis, the MDL shall be that indicated by the EPA method. For parameters that are not

TABLE 5.1 PRACTICAL QUANTITATION LIMITS (PQL) FOR VARIOUS MATRICES

MATRIX	FACTOR[1]
ground water	10
low-level soil	200
water miscible liquid waste	50
high-level soil and sludge	10,000
non-water miscible waste	100,000

[1]PQL = [PQL for ground water] X [Factor]. For non-aqueous samples, the factor is on a wet-weight basis.

Reference: USEPA SW-846, 3rd Edition, September 1986

comparable with the defined MDL in the EPA methods (i.e. laboratory cannot achieve the MDL set by the method), the laboratory MDL shall be used. For contracts/projects requiring the use of non-EPA approved methods, the laboratory shall establish detection limits according to the contract requirements and protocol.

Approach to Establishing Limits of Detection

The laboratory shall employ the procedure based on EPA Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater Appendix A (Appendix B) for the development of MDL. For contracts/project requiring non-EPA methods, the laboratory shall establish limits according to contract requirements and protocol.

6.0 DATA REDUCTION, VALIDATION, AND REPORTING

6.1 FIELD AND TECHNICAL DATA

The collected data are divided into field data and technical documentation. Technical documentation is combined field and analytical data and enables definitive characterization of the extent and magnitude of specific contaminants at each site. Field data contains data from all measurements performed on-site including well stability measurements, well logging, water level measurements, soil gas readings, and PID measurements. Technical data includes all field and analytical data plus the results of the field and laboratory QC samples and are incorporated into the final report.

Field and Technical Data Reduction

All field measurements and observations are recorded in project log books, field data records, or similar types of record-keeping books. Field measurements include pH, temperature, conductivity, alkalinity, water flow, and certain air quality parameters. All data are recorded directly and legibly in field logbooks with all entries signed and dated. If entries must be changed, the change should not obscure the original entry. The reason for the change should be stated, and the correction and explanation should be signed and dated or identified at the time the correction is made. Field data records are organized into standard formats whenever possible and retained in permanent files.

All laboratory data are cross-referenced to the appropriate trip blank, field blank, equipment blank, method blank, field duplicate or replicate, matrix spike, and matrix spike duplicate. In addition, all pertinent dates (dates collected, received by the laboratory and analyzed) for each laboratory analysis applicable to the contract or project are referenced against their respective holding times.

Field and Technical Data Validation

Validation of field data is performed on two different levels. First, all data are validated at the time of collection by following standard procedures and QC checks specified in Section 10. Second, data are validated by the Field Supervisor, who reviews the data to ensure the correct codes and units have been included. After data reduction into tables or arrays, the Field Supervisor reviews data sets for anomalous values. Any inconsistencies discovered shall be resolved immediately, if possible, by seeking clarification from the field personnel responsible for data collection. The Field Supervisor is also responsible for ensuring that defensible and justifiable data was obtained by following the field objectives described below:

- Adherence to the project work plan
- Equipment and instruments properly calibrated and in working order
- Sample collection according to standard operating procedures
- Sufficient sample volume collected to maintain sample integrity and conduct all required analyses
- Properly preserved samples
- All applicable blanks and field QC samples are provided with each sample set
- Complete COC documentation is kept throughout the duration of the field sampling effort and copies are included with each sample shipment
- Field samples arrive at the laboratory in good condition.

Random checks of sampling and field conditions are made by the Field Supervisor, who checks recorded data at that time to confirm observa-

tions. Whenever possible, peer review also is incorporated into the data validation process in order to maximize consistency between field personnel.

Once both field and analytical data have been combined, the resulting technical documentation is validated against the following criteria:

- Stated objectives of the work plan
- Stated QA objectives of the Quality Assurance Project Plan (QAPP)
- Analysis date versus the applicable holding times
- Percentage of QA analyses conducted
- Field and laboratory blank contamination
- Laboratory accuracy (percent recovery versus control limits)
- Laboratory and field precision (RPD versus control limits).

Descriptive statistic for completeness is calculated and reported.

Field and Technical Data Reporting

Description of the type and format for technical reports to be produced for the project is based on contract requirements and QAPP.

6.2 LABORATORY DATA

All bench chemists document sample preparation activities in bound laboratory notebook. These serve as the primary record for subsequent data reduction. The data for GC/MS, AA, ICP, and GC analyses are generated by stand-alone computers. The data for Mercury analysis is conducted using a strip chart to record absorbance expressed in peak height units. Results of each analysis are transcribed manually onto analytical results forms specific to the particular analysis. All data are checked for accuracy and precision at the bench and instrument operator/analyst level, the laboratory manager's level and the QAO's level. The QAO's review shall consist of comparing spike recovery and/or relative percent difference to control limits established for the parameter analyzed. Concentration of the analytes found in the analysis are expressed according to the required units, depending on the sample matrix (i.e., ug/L for aqueous samples for ug/Kg for soil samples).

Laboratory Data Reduction

Gas Chromatograph Results—Calculations are performed for each analyte after its identification is determined. Identification is based on retention time of the suspect peak compared to the retention time of the internal standard. The concentration of the analyte is determined by using the calibration curve and the peak area of the analyte. A response factor is determined from the calibration curve and used to calculate the concentration. Final results will be rounded to the nearest 0.1 ug/L (or two significant figures, whichever is smaller). (See Appendix D, Significant Figures)

Gas Chromatograph/Mass Spectrometry Results—Qualitative identifications are determined by obtaining extracted ion current profiles (EICPs) for the primary ion mass to charge ratio (m/z) and the secondary masses for each analyte. Positive identification is based on the following criteria:

- The intensity of the three characteristic masses of each analyte must maximize in the same ratio (±20%), within one scan of each other
- The retention time must fall within ± 30 seconds of the retention time of the authentic compound
- The relative peak heights of the three characteristic masses in the EICPs must fall within ± 20 percent of the relative intensities of these masses in a reference mass spectrum (standard analysis or reference library).

Structural isomers to be listed as separate analytes must have an acceptable resolution. Acceptable resolution is achieved if, in a standard mix, the baseline to valley height between the isomers is less

than 25 percent of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

The calculation for the concentration for the suspect peak is made using the RF for each analyte.

$$\text{Concentration} = \frac{(A_s)(C_{is})}{(A_{is})(RF)}$$

where:

A_s = Area of characteristic m/z for the analyte to be measured
A_{is} = Area of characteristic m/z for the internal standard
C_{is} = Concentration of the internal standard, in $\mu g/L$
RF= Average response factor as calculated from the area formed on an intensity plot of the ion of interest.

Inductively Coupled Plasma (ICP)—The theory of emission spectrometry is based upon excitaton of gaseous atoms of an element resulting in the electrons being raised to high energy state. The frequency of the emitted radiatoin depends on the difference in energy of the two states of the atom. While the intensity is relative to the concentration of the element.

$$E_2 - E_1 = hv = hc/w$$

E2 = to the energy of the higher excited state
E1 = to the energy of the ground state
h = Planck's constant
v = frequency of emitted light
c = speed of light
w = wavelength (lambda)

The purpose of the ICP is to measure concentrations of elements in a sample. This is accomplished by measuring the emission intensity produced when a sample containing these elements is aspirated into the plasma exciting the electrons to a higher state. The measurement of intensity if performed by allowing the light leaving the plasma through each exit slit to fall on a photomultiplier tube. This tube converts light energy into an electrical current. At the end of the integration time the total current produced is measured and is proportional to the intensity and the concentration of the element being analyzed. This total current value is sent to the computer for data reduction. The final calculations are done by the computer system by comparing intensity of unknown against intensity of known standards.

Interelement correction is performed by measuring the light intensity of interfering elements and mathematically correcting for the additional emitted intensity.

Atomic Absorption Spectrophotometry Results—Photometric absorbance is governed by the relationship:

$$\text{Absorbance} = \log (100/\%\,T) = 2 - \log \%\,T$$

where:
% T = 100 - % absorption

Percent Absorption is based on the amount of light of a particular wavelength absorbed by a specific metal. Its calculation is based on the loss of light after a beam of light of a particular wavelength is passed through a flame into which a solution containing metals of interest has been aspirated.

Calibration curves establishing the absorbance relationship with concentration are generated at various concentrations. From these curves, a comparison is made with absorbance from sample measurement. Since absorbance is directly related to concentration, a plot of the two parameters is linear in certain operable ranges and allows for determination of unknown concentrations in solutions (direct samples or extracts) after measurement of absorbance.

Atomic absorption spectrophotometry is based on the principle that if light of a resonance wavelength is passed through a flame containing atoms of an element to be measured, then part of the light is absorbed and the extent of absorption is proportional to the number of atoms present in the flame. Because of the sophistication of current instrumentation, the partial application of this technique for the measurement of metal concentration in liquids relies on a Beer's absorption law approach, comparing absorbance from an unknown against the linear correlation between absorbance and concentration in standards.

In many spectrophotometric measurements, interferences occur in the absorption of light confusing

the Beer's Law relationship between absorption and concentration; this is especially true for atomic absorption. To alleviate this problem, a technique known as the "method of standard additions" is used in which sequential known amounts of the component being analyzed for are added to a sample of unknown concentration. By making an initial absorbance measurement and measurement after each addition, the effects of interference present in the analytic matrix can be accounted for.

Laboratory Data Validation and Data Reporting

Data validation is performed by the QA Officer and the PO. Validation is accomplished through routine audits of the data collection and flow procedures and by monitoring of QC sample results.

Validation Requirements

The minimum requirements for each analytical run are:

- At least three point calibrations and one calibration check standard
- Continuing calibration check using an EPA or NBS reference, if available
- Laboratory control samples/QC sample included in each analysis whenever available
- One reagent blank per matrix and per concentration level for every sample batch analyzed (i.e., one per 20 samples)
- Matrix spike duplicate and matrix spikes per concentration level and per matrix for every sample batch analyzed (i.e., one set per 20 samples)

For GC/MS data, in addition to the requirements mentioned:

- DFTPP or BFB key ions and abundance criteria
- Percent recovery of surrogate spike run on all samples
- Internal standard area monitoring for samples analyzed (-50% to +100%) of the internal standard area of the calibration standard
- RIC/Quan, standard curves and printouts of samples analyzed

Data Collection and Flow Audits

Data collection and flow audits include the following:

- Review of sample documents for completeness by the analyst(s) at each step of analysis
- Daily review of instrument logs, performance test results, and analyst performance by the laboratory manager
- Daily review of performance indicators such as blanks, surrogate recoveries, duplicate/matrix spike duplicate analyses, matrix spike analyses, etc.
- Random calculation checks
- Review of all reports prior to and subsequent to data entry
- Review and approval of the final report of the QA Officer

Data Review

The review of data quality involves several levels of evaluation. In general, the analysts and the laboratory manager are responsible for reviewing the data relative to instrument calibration, standard preparation, method blanks, raw data, calculations and transcriptions. The analyst normally reviews 100% of the raw analytical data generated, including the calibration data and all calculations. Upon completion of the analyst process, a second level of review of the raw data is performed by either the laboratory manager or Program Manager. At this level, 25 - 100% of the data quality indicators (i.e., method blanks, replicate analyses and spike recovery data) are reviewed relative to the acceptance criteria described in the analytical procedures.

At the next level of data quality review, the QA Officer is generally responsible for a complete review of about 20% of the data generated. The emphasis is on the data acceptability relative to the data quality indicators and on the accuracy of the final data summaries.

All analytical problems encountered during sample analysis are properly addressed to provide explanations for data users.

Review Checklists

See figure6-1 and 6-2.

REPORT REVIEW

CLIENT: **PROJECT:** **SDG/EPISODE:**

ITEM	CHECK	✓	REMARKS/CORRECTIONS
Case Narrative	Typographical		
	Names		
	Ref IDs/Text IDs		
	Receipt Dates		
	Single/Plural Spl		
	VOA-Gold Sheet		
	BNA-Extract Log		
	QC Inorg Notes		
Chain of Custody	Original		
	Copy		
Cooler Receipt Form	Yes		
	No		
Report - Headers	Dates		
	Date Sampled		
	Date Extracted		
	Date Analyzed		
	Sample IDS		
	Client		
	Lab		
	Proj Ref Consistant		
	Method Reference		
	Dilution Factors		
Report - Data Review	Units of Measure		
	Values Transcribed		
	Flags Correct		
	Surrogate Recoverie		
	% OK		
	Range to Matrix		
QC Section	Header Information		
	Units of Measure		
	Values Transcribed		
	Surrogate Recoverie		
	% OK		
	Range to Matrix		
Diskette	CBDF		
	Directory CK		
	Detection Lmts		
	Spike Amounts		
	QC Check		
REVIEWER:	DATE:		CORRECTED (Initials/Date):

Instructions

A check mark in the appropriate column indicates that the reviewer has reviewed and verified the item next to it. If a check is not marked please refer to "REMARKS/CORRECTIONS column for report numbers which need correction or additional comments from the reviewer. CORRECTED Initials/Date indicate that corrections were made and verified.

Figure 6-1 Report Review Sheet

DATA REVIEW CHECKLIST

PROJECT OFFICERS DEPARTMENT

CLIENT/PROJECT: _____

EPISODE: _____

REVIEW DATE: _____

Client Name/Address/Contact are all correct?	Yes ☐	No ☐
Sample I.D.s are as specified on client chain-of-custody?	Yes ☐	No ☐
If No: Is the I.D. change documented in a client phone log and in the case narrative?	Yes ☐	No ☐
Do all samples reflect the proper matrix?	Yes ☐	No ☐
Are all units reported reflecting the proper matrix?	Yes ☐	No ☐
Are the proper method references listed?	Yes ☐	No ☐
Do the results on the worksheets agree with the LIMS entered result?	Yes ☐	No ☐
Do the results from the raw data agree with reported results?	Yes ☐	No ☐
Are Dilution Factors/Calculations carried out correctly?	Yes ☐	No ☐
Are all parameters addressed in the QC section?	Yes ☐	No ☐
Do they have the following QC information tabulated?		
1. Method &/or TCLP Blank Results?	Yes ☐	No ☐
2. Blank Spike Results?	Yes ☐	No ☐
3. Laboratory Control Spike (LCS)?	Yes ☐	No ☐
4. Matrix Spike/Matrix Spike Duplicate Results?	Yes ☐	No ☐
5. Duplicate Sample Results?	Yes ☐	No ☐
Are Diskette Deliverables Required?	Yes ☐	No ☐
If Yes:		
Is the requested format supplied?	Yes ☐	No ☐
Are all the fields filled out?	Yes ☐	No ☐

Are CLP/"CLP-Like" Packages required by client?	Yes ☐	No ☐
If Yes: Are the following Items supplied?		
ORGANICS:		
1. QC Summary (i.e., Surrogate, MatrixSpike/ Matrix Spike Duplicate Summary, Method Blank Summary)	Yes ☐	No ☐
2. Sample Results?	Yes ☐	No ☐
3. Sample raw Data?	Yes ☐	No ☐
4. All Pertinent Calibration Summaries (Initial & Continuing)?	Yes ☐	No ☐
5. All Pertinent Calibration Raw Data?	Yes ☐	No ☐
6. All Tuning Summaries (BFB & DFTPP)?	Yes ☐	No ☐
7. All Tuning Raw Data?	Yes ☐	No ☐
8. Method Blank Raw Data?	Yes ☐	No ☐
9. MS/MSD Raw Data?	Yes ☐	No ☐
INORGANICS:		
1. Sample Results?	Yes ☐	No ☐
2. QC Summary (i.e., Initial & Continuing Calibration Verification, CRDL Standard for AA & ICP, Blanks, ICP Interference Check Sample, Spike Sample Recovery, Post Digestion Spike Sample Recovery, Duplicates Laboratory Control Sample, Standard Addition Results, ICP Serial Dilutions, Preparation Log, Analysis Run Log)?	Yes ☐	No ☐
3. Quarterly Verification of:		
Instrument Parameters?	Yes ☐	No ☐
Instrument Detection Limits?	Yes ☐	No ☐
ICP Interelement Correction Factors?	Yes ☐	No ☐
ICP Linear Ranges?	Yes ☐	No ☐
4. Raw Data?	Yes ☐	No ☐

ADDITIONAL COMMENTS:

_____

_____

_____

_____

Project Officer Signature: _____

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[P]007-0692-05]

Figure 6-2 Data Review Checklist

7.0 EXTERNAL AND INTERNAL QUALITY CONTROL CHECKS

7.1 EXTERNAL QUALITY CONTROL CHECKS

EPA CLP Performance Evaluation and On-Site Audits

Quarterly Performance Evaluation (PE) samples consisting of one to three sets of PE samples are analyzed by the laboratory to demonstrate its ability to perform CLP requirements. This provides evidence that the laboratory personnel involved fully understand the required analytical methods and that these methods can be performed satisfactorily by the laboratory personnel using the laboratory equipment and instrumentation. It also provides evidence that the laboratory understands the documentation and reporting requirements of the contract.

An on-site audit is done annually to demonstrate the laboratory has in place and operating, all the personnel, equipment and internal standard operating procedures needed for performance of contract requirements.

EPA Water Pollution (WP) /Water Supply (WS) Performance Evaluation

The laboratory receives sets of quarterly PE samples for the analyses of water pollution and water supply parameters to provide interlaboratory evaluation of data results for reproducibility and comparability. It also provides "feedback" of the QC procedures used by the laboratory.

Performance Evaluation of Contracts/Project Proposals

A set of PE samples is received by the laboratory prior to pre-award of contract/project proposal bids. PE results obtained demonstrate the data reliability of sample analyses performed under a contract/project.

Field Quality Control Checks

Collection and analysis of field blanks, equipment washes, trip blanks, and field replicates are intended as QC checks on the integrity of sample collection and handling procedures and equipment decontamination procedures.

Field blanks, equipment blanks, and trip blanks are prepared using ASTM Type II reagent water and sample bottles randomly selected from the bottles prepared for environmental samples. ASTM Type II reagent water is used to prepare these field check samples, regardless of the environmental medium being sampled, because:

- It mimics the physical characteristics of groundwater and surface water
- No reproducible, affordable material is available that mimics the clay and organic portion of soils and sediments
- An organic or aqueous reservoir is necessary for the absorption, dissolution, or solvation of organic and inorganic contaminants.

Field blanks (ambient conditions blanks) are prepared at the beginning of each sampling event, at each discrete sampling site, by pouring ASTM Type II reagent water into prepared sample bottles. These sample bottles are randomly selected from the supply of prepared sample bottles; a sample container is selected that is appropriate for each type of analysis for which environmental samples are being collected. The field blanks are handled and analyzed in the same manner as environmental samples. Because field blanks and environmental samples are collected under the same conditions, field blank analyses are used to indicate the presence of external contaminants that may have been introduced into samples during collection. Field blanks also may become contaminated during transport, but this may be assessed by the simultaneous use of trip blanks, which is discussed below.

Equipment blanks (bailer washes) are prepared for manual and small automated sampling equipment used to collect environmental samples (i.e., equipment blanks are not prepared for drill rig sampling equipment). Equipment blanks are collected during the sampling day by pouring ASTM Type II reagent water into/through/over a clean piece of sampling equipment, such as bailers, shovels and trowels, and then dispensing it into prepared sample bottles. These sample bottles are randomly selected from the supply of prepared sample bottles, selecting a sample container appropriate for each type of analysis for which environmental samples are being collected. Analyses of bailer washes are used to assess the efficiency of equipment decontamination procedures in preventing cross-contamination between samples.

Trip blanks are prepared at the beginning of the sampling trip by pouring ASTM Type II reagent water into prepared sample bottles. These sample bottles are randomly selected from the supply of prepared sample bottles. Sample containers are filled to yield an appropriate sample volume for each type of VOC analysis, resulting in a complete trip blank for the sampling event. These trip blanks are prepared at the laboratory, shipped to the facility to be sampled (with the unused sample bottles), stored with the unused sample bottles, transported to the sampling site, and then shipped for analysis with the samples collected during the sampling event. The water used to prepare each batch of trip blanks is analyzed for VOCs. The results of the trip blank analyses are reported along with the associated environmental and QC samples. The trip blanks remain unopened throughout the sampling event. Analysis of trip blanks is used to assess contamination of sample containers during transport to and storage at the site, and contamination of samples during transport back to the laboratory. One trip blank will be included in each shipping container containing samples for VOC analysis.

All sample containers provided are shipped with COC records. These records are completed by the field personnel and returned with the samples. The following QC samples are collected for each day of sampling:

- One trip blank per cooler per sampling team for every batch of VOC samples.
- One field blank per field sampling team for every VOC sampling round at a particular site or zone.
- One set of equipment blanks for every day of groundwater sampling. All parameters are to be analyzed.
- One field replicate for every 20 soil/sediment samples is collected at a preselected monitoring point. (More frequent replication can be accomodated.) Field replicates are collected at the same time and in the same manner as the other environmental samples. Field replicates are not the same as laboratory duplicates, because they are separate samples obtained from the same field monitoring point. As such, results of the field replicate analyses are used to assess the precision of the field sampling methods, not that of the analytical techniques.
- One field duplicate for every 20 water samples is collected at a preselected monitoring point. (More frequent replication can be accomodated.) A duplicate sample is collected independently at a sampling location during a single act of sampling. Field duplicates are labeled indistinguishable from other analytical samples so that personnel performing the analyses are not able to determine which samples are duplicates. Field duplicates are used to evaluate the reproducibility of sampling techniques.

7.2 LABORATORY QUALITY CONTROL CHECKS

Matrix Spike/Matrix Spike Duplicate (MS/MSD)

MS/MSD is used to check for precision and accuracy. These are replicate samples spiked with a known spike concentration that are taken through the whole sample preparation process. MS/MSD analyses are performed on one sample in each group of 20 samples (20%), and/or on each type of sample matrix per concentration. Table 7.2 shows a list of matrix spike compounds routinely employed by the laboratory.

The sample analysis process and the spike sample process differ in the adding of known amounts of the substances to be analyzed to the aliquot of the replicate sample. The amount of spike added varies according to the working range of the analytical instrument.

After value of the sample is determined, the value of the sample spike is determined. Should the sample also have a value of the spike analyte, the value of the sample is subtracted from the value of the spike and the percent (%) recovery of the spike is calculated using the following equation:

$$\% \text{ Recovery} = \frac{[\text{Spike Sample Result} - \text{Sample Result}] \times 100}{\text{Spike Added}}$$

At times the sample value is outside the operating range of the analytical instrument. In such cases it is impossible to know the magnitude of the analyte before the spike is added. Occasionally, the sample and the spike sample require dilution to perform sample analysis within the linear range of the instrument. This dilution adjusts the analyte in the sample to the proper concentration, but it will sometimes also dilute the spike added below the range of detection. In this case, it is not possible to report spike recovery for that particular analyte.

The lack of spike recovery data for an analyte that has been diluted to levels outside the working concentration of the instrument is supplemented by the periodic analysis of spiked QC check sample and other additional sample data.

The calculated % recoveries are then used to assess data precision expressed as relative percent deviation (RPD). It is calculated using the following equation:

$$\text{RPD} = \frac{[\text{MS Result} - \text{MSD Result}] \times 100}{\text{Mean of MS and MSD Results}}$$

where MS denotes Matrix Spike.

RPD can also be measured by duplicate sample analysis of one sample. In this case the (2) sample results take the place of the MS and MSD values in the above equation.

Method Blanks

Method blanks, also known as reagent blanks, are analyzed for each matrix and each batch of sample analyses. An aliquot of equal volume or weight to the sample is used for method blank analysis. The method blank, like that of duplicate and spike samples, is taken through the whole analytical process. The method blank must be blank of any substances being analyzed, or interferences.

Calibration Blank/System Blank

A calibration blank/system blank is prepared by analyzing the same matrix used for the preparation of the calibration standards. It is used to establish the analytical curve by taking into account background responses during the calibration process. It is also used to check for carry-over contamination after a standard run or after a contaminated sample run.

Laboratory Control Samples (LCS)

Laboratory Control Samples (LCS) are obtained from USEPA Quality Assurance Branch, Environmental Monitoring and Support Laboratory (EMSL), and NBS. The quality control samples are prepared for analysis in strict accordance with the procedures provided with the materials. LCS are analyzed each day for every sample batch run to check proficiency of the analysis in terms of working standards preparation, monitor standard degradation, and check traceability of the prepared standards from commercial reference materials to EPA reference materials. A measure of comparability between batches is established with the analysis of the LCS. It is also used to check efficiency of both the digestion/extraction, and the instrumental analysis. Check results must be within the target range values (i.e. 95% confidence limits of the given values) of the LCS or within the 95% confidence limits of the true value of the check standard as determined from running replicate analyses of the check standard. (See Appendix D for Standard Traceability)

Surrogate Spike Analyses

Where applicable, an analytical process includes the addition, subsequent detection, and recovery calculation of surrogate spiking compounds. Surrogate compounds are analyte compound substitutes, (i.e., compounds not specifically requested to be determined as analytes in a particular scope of work) which most often do not occur naturally. Surrogate compounds are added to samples for analysis after sample aliquots have been measured out and are taken through the whole sample preparation process. Surrogate compounds, to be useful in QC analysis, must not interfere with the determination of the analytes of interest. Surrogates must also be chemically similar to the analytes of interest and capable of emulating the analyte response.

Surrogate standard determinations are performed on all samples and blanks. All samples and blanks are spiked with surrogate spiking compounds before purging or extraction in order to monitor preparation and analysis of samples. Table 7.1 shows a list of surrogate spike compounds routinely employed by the laboratory.

Method of Standard Addition

A method of standard addition is used to check the accuracy of the analysis method under optimum conditions, excluding chemical interference from sample matrix. (See Section 4.2.1.1 for details)

ICP Interference Check Sample (ICS) Analysis

ICP interference check sample verifies interelement and background correction factors of the ICP instrument. (See Section 4.2.1.1 for details)

EPA, NBS and/or Commercial Reference Standards

These standards are analyzed routinely for the parameter of interest. Commercial standards are checked for standard traceability.

Internal Standards Analysis

Internal standard areas are monitored as a measure of instrument calibration. Internal standard determinations are performed on all samples and blanks to monitor instrumental efficiency and also used as a reference retention time indicator to check retention time shifts of peaks of interest.

A known amount of internal standard concentration is added to a sample extract prior to instrumental analysis. Like the surrogate standard, it must not interfere with the determination of the analytes of interest. It must also be chemically similar to the analytes of interest and capable of emulating the analyte response.

Calibration Standards

Calibration standards are used to quantitate the amount of analytes present in the sample. These are analyzed to initiate any type of analyses. (See Section 4.0, Calibration and Frequencies)

Tuning Requirements (BFB and DFTPP)

See Section 4.5, Table 4.2, and Table 4.3.

Control Charts

The performance of a measurement system can be demonstrated by the measurement of homogeneous and stable control samples in planned repetitive process. The data generated is plotted as a control chart to indicate whether the system is measuring the control sample adequately to provide confidence in the measurement process. It warns the laboratory of possible deviation from 95% confidence level by identifying systematic errors, drifts, or other types of problems.

The use of control charts are summarized as follows:

■ They provide graphic assessment of accuracy and precision for the analysis of each analyte and instant detection of erroneous data.

■ They allow efficient observation of recovery trends for a particular analysis, and they provide a long-term mechanism for self-evaluation of analytical output.

■ They provide assessment of the analytical capability of the staff chemists with regard to the output of valid analytical data.

■ They allow observations of deviations from control trends. It has been noted that:

√ A system must be established to be in control, in order to be maintained in control.

√ A system is not in control if it is observed to produce unexpected data more than once every 20 runs.

√ Control limits usually become tighter once a process is under a controlled protocol (i.e. the original limits were based on data produced by an uncontrolled operation).

TYPES OF CONTROL CHARTS USED

Four charts are used to monitor the laboratory data:

Surrogate Spike Recoveries vs. Sample Analyzed (see fig 7.1)

Laboratory Control Standard (LCS) Concentration vs. Date Analyzed (see fig 7.2)

Percent Matrix Spike Recovery vs. Date Analyzed (see fig. 7.3)

Relative Percent Difference of Matrix Spike/MS Duplicates or Duplicate Analyses vs. Date Analyzed (see fig. 7.4)

The preparation of a control chart is based on Shewhart's theory of control charts (EPA Handbook for Analytical Quality Control Water and Wastewater Laboratories, Chapter 6).

APPROACH TO CONTROL CHART INTERPRETATION

For each parameter and method, a data base of percent recovery (%R) for QC reference samples or spiked samples is collected. The arithmetic mean and standard deviation of this set is calculated. From this information, warning and control limits of a run are determined. These are defined as:

■ Warning limits are defined as x ±2s, where s is standard deviation.

■ Control Limits are defined as x ±3s, where s is standard deviation. The %R of each QC sample or spike sample is plotted on a control chart and compared with the statistically based control limits.

■ Data precision is evaluated based on results of the samples analyzed in duplicate. The range is calculated and then divided by the average of the two analyses, then multiplied by 100. This value equals percent difference (%D). The %D of duplicates in each data set is compared with the values previously found in the laboratory. Calculations for warning and control limits are the same as above.

■ Interpretation of control charts for out-of-control:

■ One or more points outside the control limit (3s).

■ A run of two or more consecutive points outside warning limits (2s).

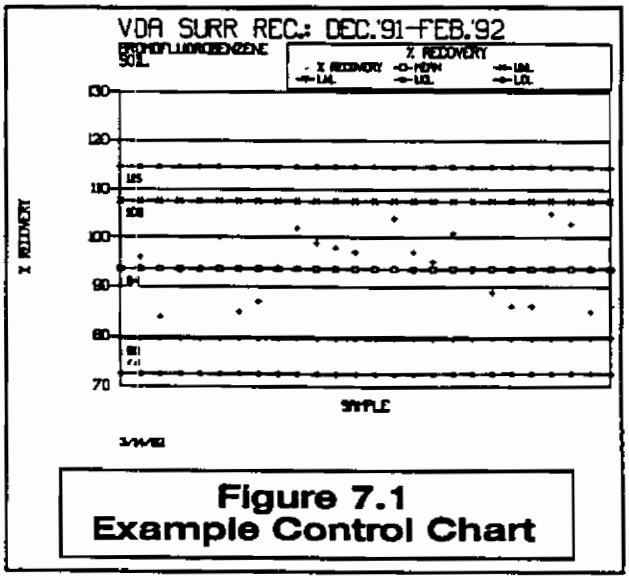

Figure 7.1
Example Control Chart

■ A run of seven or more points above or below x, indicating trends or shifts.

■ Cycles or non-random patterns in the data chart (six consecutive points increasing or decreasing).

■ See figure 5.4 for examples of some of these outliers/trends.

■ Control charts are routed to the various departments where trends are noted and corrective actions are made to alleviate these patterns.

Laboratory Control Limits

Appendix E shows table of laboratory control limits for some parameters. Data outside of the laboratory control limits are flagged and discussed with the client. As the laboratory gathers a large enough data base, it will be able to establish its own criteria for 95% confidence level for both inorganic and organic parameters.

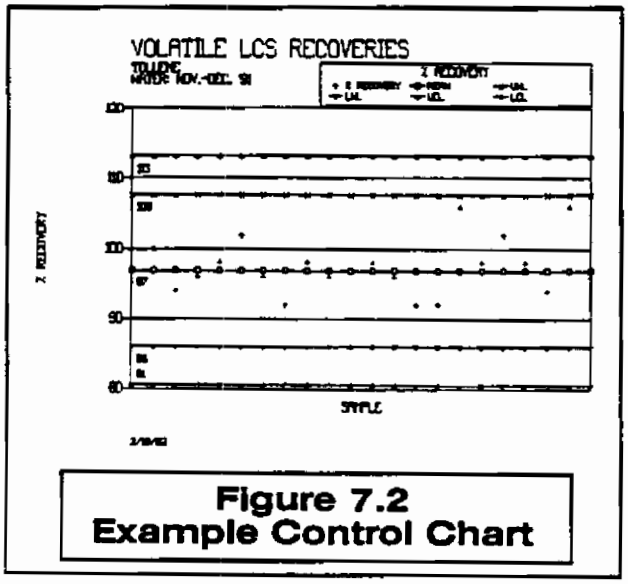

Figure 7.2
Example Control Chart

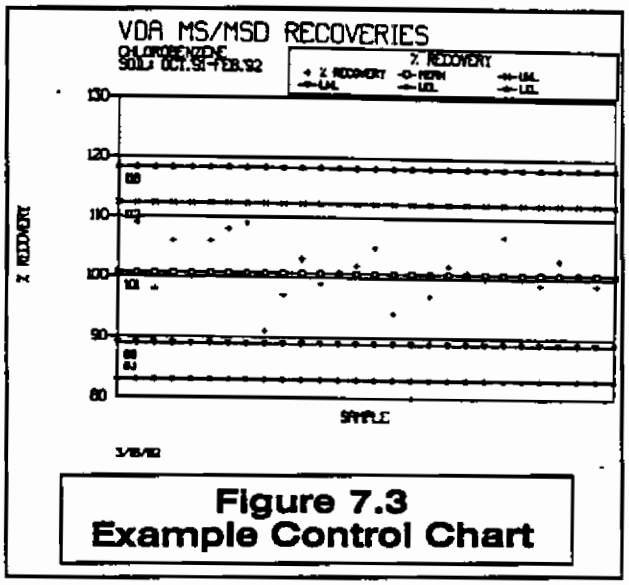

Figure 7.3
Example Control Chart

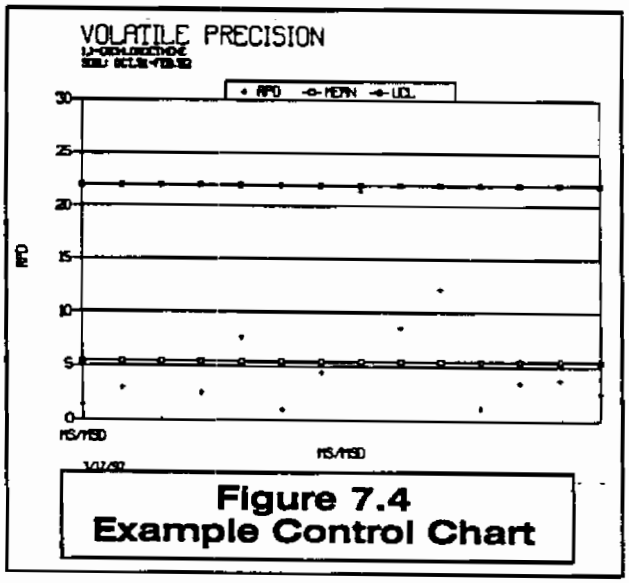

Figure 7.4
Example Control Chart

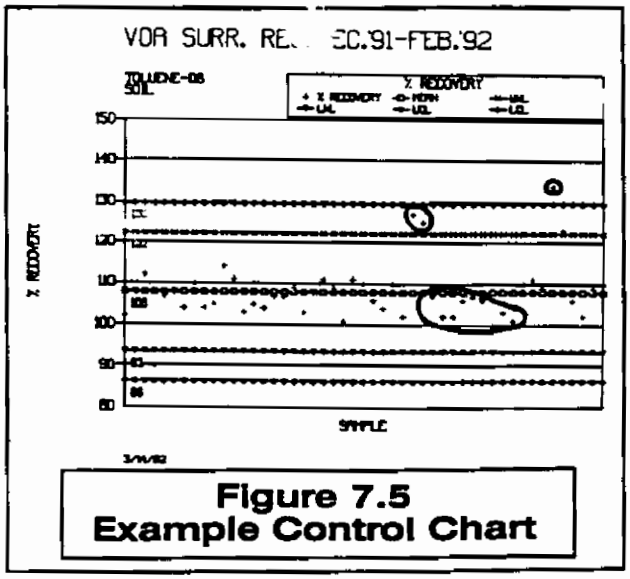

Figure 7.5
Example Control Chart

TABLE 7.1 LIST OF SURROGATES SPIKING STANDARDS FOR ORGANIC ANALYSIS

SURROGATES

Method 601/602/8010/8020 (GC Purgeables):
- Bromochloromethane
- Bromofluorobenzene

Method 608/8080/IFB (Pest/PCB)
- Dibutyl Chlorendate (DBC)

Method 624/8240/IFB (GC/MS Purgeables)
- 1,2 Dichloroethane-d4
- Bromofluorobenzene
- Toluene-d8

Method 625/8270/IFB (Semivolatiles)
- Nitrobenzene-d5
- 2-Fluorobiphenyl
- 2,4,6-Tribromophenol
- Phenol-d5
- 2-Fluorophenol
- Terphenyl-d14

TABLE 7.2 LIST OF MATRIX SPIKING STANDARDS FOR ORGANIC ANALYSIS

MATRIX SPIKES

Method 601/602/8010/8020 (GC Purgeables)
- 1,1-Dichloroethene
- Trichloroethene
- Chlorobenzene
- Toluene
- Benzene

Method 608/8080/IFB (Pest/PCB)
- Gamma-BHC
- Heptachlor
- Aldrin
- Endrin
- PP'-DDT
- PCB-1260 or PCB-1254

Method 615/8150 (Herbicides)
- 2,4-D
- 2,4,5-TP (Silvex)

Method 624/8240/IFB (GC/MS Purgeables)
- 1,1-Dichloroethene
- Trichloroethene
- Chlorobenzene
- Toluene
- Benzene

Method 625/8270/IFB (Semivolatiles)

BASE/NEUTRALS	ACIDS
1,2,4-Trichlorobenzene	Phenol
Acenaphthene	Pentachlorophenol
2,4-Dinitrotoluene	4-Chloro-3-Methylphenol
Pyrene	2-Chlorophenol
N-Nitroso-di-N-Propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

8.0 PERFORMANCE AND SYSTEM AUDITS

Audit is defined as systematic check to determine the quality of operation of laboratory activities. It is comprised of the following:

- Performance audit
- System audit

8.1 FIELD PERFORMANCE AUDITS

Field performance audits are performed on an ongoing basis during the project as field data are generated, reduced, and analyzed. All numerical analyses, including manual calculations, are documented. All records of numerical analyses are legible, of reproduction-quality, and supporting complete to permit logical reconstruction by a qualified individual other than the originator.

Other indicators of the level of field performance are the analytical results of the blank, duplicate and replicate samples. Each blank analysis is an indirect audit of effectiveness of measures taken in the field to ensure sample integrity (e.g., field decontamination procedures). The results of the field duplicate and replicate analyses are an indirect audit of the ability of each field team to collect representative sample portions of each matrix type.

8.2 LABORATORY PERFORMANCE AUDIT

Procedures used to assess the effectiveness of the quality control system are as follows:

Internal Performance Audits

These are accomplished by the laboratory through the use of control samples, replicate measurements and use of reference materials.

Sample analysis systems are conducted by the QA Officer and include the following:

- Verification of written procedures and analyst(s) understanding
- Verification and documentation of procedures and documents
- Review of analytical data and calculations

External Performance Audits

These are accomplished by the laboratory through interlaboratory checks such as:

- Participation in various state laboratory evaluation programs.
- Participation in WP & WS studies from EPA.
- Participation in Environmental Resource Assoc (ERA) WP & WS studies.
- Analysis of split samples and comparing results with the other laboratory.
- Participation in the U.S. EPA CLP program.
- Participation in the U.S. Army Corps of Engineers DERA certification program.

8.3 LABORATORY SYSTEM AUDIT

An on-site inspection is done by EPA audit team or laboratory certification personnel to review the laboratory quality control system which covers sample handling, sample analysis, records control, preventive maintenance, and proficiency testing. When EPA or laboratory certification personnel initiate a system audit of the laboratory, any recommendations made or deficiencies identified will be considered for implementation and corrective actions taken to correct deficiencies.

LABORATORY AUDIT CHECKLIST

QA/QC DEPARTMENT

DATE: _____

BY: _____

SAMPLE RECEIVING:

1. Was the sample paperwork & laboratory paperwork in agreement?* Yes☐ No☐

2. Was a sample log-in sheet filled out? Yes☐ No☐
   If so, were all items completed? Yes☐ No☐

3. Was the worksheet and appropriate paperwork sent out to the analytical requred sections of the laboratory (i.e., Extractions, Gc/MS, & Inorganics)? Yes☐ No☐

4. Were there any revisions made to the paperwork and notes/phone logs describing why the revisions were made? Yes☐ No☐

5. Were samples storage areas monitored for appropriate temperatures? Yes☐ No☐

6. Any temperatures outside of control limits? Yes☐ No☐
   If so, were corrective actions made? Yes☐ No☐

COMMENTS:

*Make copy of paperwork to verify that it is in agreement with other parts of the laboratory.

ANALYTICAL LABORATORIES:

1. Does log-in of sample/extracts to the analytical lab agree with the Sample Receiving paperwork (i.e., analytical run logs I.D. samples correctly)? Yes☐ No☐

2. Are samples analyzed within holding times? Yes☐ No☐

3. Are standards used in analysis traceable back to US EPA standards? Yes☐ No☐

4. Are calibrations performed correctly? Yes☐ No☐

5. Is the analytical run sequence in agreement with the requested methodology? Yes☐ No☐

6. Were there any corrective actions/maintenance performed to the instrument during analysis of the samples in question? Yes☐ No☐

7. Are these corrective actions/maintenance documented in the instrument maintenance logs? Yes☐ No☐

COMMENTS:

REPORTING:

1. Does the Sample Receiving paperwork agree with the reporting paperwork (i.e., sample number, sample ID's, turnaround time, matrix, etc.) Yes☐ No☐

2. Have revisions been made to the paperwork? Yes☐ No☐

3. Are notes/phone logs describing revisions included in the reporting files? Yes☐ No☐

4. Are problems in the analysis reported in the case narrative?* Yes☐ No☐

5. Was the reporting requirements in agreement with what the client requested? Yes☐ No☐

6. Were there any resubmissions or submissions of late data? Yes☐ No☐

7. Were the reasons for these submissions documented in the subsequent case narratives? Yes☐ No☐

8. Were requested methodologies reported to the client? Yes☐ No☐

9. Was all appropriate QC (Quality Control) reported? Yes☐ No☐

COMMENTS:

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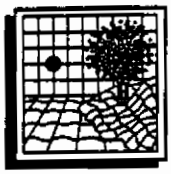
FIGURE 8-1 Laboratory Audit Checklist



LABORATORY CORRECTIVE ACTION

QA/QC DEPT.

➤ Contract/Project/Episode Number:

DESCRIPTION OF EVENT

➤ Date Recognized: ➤ By:

➤ Date Occurred: ➤ By:

➤ Date Corrected: ➤ By:

ANALYTICAL METHOD

➤ Analyte: ➤ Method:

➤ Analyst: ➤ Lab Manager:

➤ Samples Affected (Lab ID):

➤ Description of problem encountered:

➤ Corrective/Preventive Action Taken:

➤ **SIGNATURES:**

Analyst: *Date:*

Supervisor *Date:*

QA Officer *Date:*

FOLLOW UP

➤ Follow up Investigation/Discussion:

➤ **SIGNATURES:**

QA Officer: *Date:*

Laboratory Director: *Date:*

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FIGURE 8-2 Laboratory Corrective Action

9.0 PREVENTIVE MAINTENANCE

Preventive maintenance is defined as an orderly program of positive actions for preventing failure of equipment and ensuring that the equipment is operating with the reliability required for quality results. The actions include specification checks, calibrating, cleaning, lubricating, reconditioning, adjusting and checking.

9.1 FIELD EQUIPMENT

Preventive maintenance is carried out on all field equipment prior to being shipped to the sampling site. This preventive maintenance includes regular battery checks and maintaining a sufficient stock of spare parts and supplies. Field personnel are strongly cautioned that these instructions are for general purpose only. Should equipment break down in the field and field crews are unable to repair the equipment within a reasonable amount of time, the operations manager in the laboratory is notified. A replacement is shipped immediately via overnight courier. Whenever possible, duplicates of all equipment are initially sent to the field. For specific preventive maintenance procedures, the appropriate instrument manual should be consulted.

pH Meter

The following is a description of the preventive maintenance procedures for the field pH meters:

- Charging Batteries

After the initial charge when first placing the instrument in operation, the batteries are recharged after each 30 hours of operation. Allow the batteries to charge 16 hours to restore them fully. Exceeding the 16-hour period will not damage the batteries. Overnight charging is recommended, and periods of operation between charges should not exceed 30 hours. With proper charging practices, a set of batteries should last for more than 300 charge cycles.

- Battery Replacement

When batteries no longer hold a charge for a reasonable length of time, they should be replaced. This unit requires six AA size nickel-cadmium batteries. Replace them as follows:

- Remove the accessories from the foam insert above the instrument panel
- Remove the four screws securing the panel in the case
- Lift the panel from the case and place it face down on a padded surface
- Pry the batteries from their clips with a screwdriver and replace all six batteries
- Replace the panel in the case, and replace the accessories in the foam insert
- Connect the charge unit to the instrument and allow the batteries to charge for 15 hours.

- pH Electrode Care Storage

When the electrode is not in use, the wetting cap with filling solution-soaked cotton should be reinstalled over the tip, and the fill hole cover should be placed over the hole. This will prevent loss of filling solution through evaporation. Always maintain the filling solution level just below the fill hole.

- pH Electrode Cleaning

Normal cleaning of the electrode can be performed in the following manner:

- Immerse the electrode tip in 0.1N HCl followed by immersion in 0.1N NaOH and again in 0.1N HCl, each for a 2 minute period. Rinse with ASTM Type II reagent water and soak in pH 7 buffer solution for 30 minutes.
- If the electrode is slow to respond or readings are unstable and the connection cannot

be remedied with normal cleaning, the reference junction may be clogged. Clean the junction for 10 minutes in dilute potassium chloride solution. First dilute a saturated potassium chloride solution about 1:10 with water. Place the electrode tip in the boiling solution for about 10 minutes.

• Remove heat and allow the electrode to cool while immersed in the solution. Then rinse with ASTM Type II reagent water and soak in pH 7 solution before testing again.

If these steps fail to improve electrode response, replace the electrode. If the pH bulb becomes contaminated or left dry, it may be reconditioned by following the cleaning procedure above.

Specific Conductance Meter

The following is a description of the preventive maintenance procedures for the field specific conductivity meters:

- Battery Replacement

Low battery condition is indicated by an arrow on the display. When the arrow appears, the battery should be replaced.

Thermometer

After each use, the thermometer probe should be rinsed with ASTM Type II reagent water. Should the sample contain oils or other heavy hydrocarbon mixture, the probe should be washed with laboratory-grade detergent and rinsed with ASTM Type II reagent water.

9.2 LABORATORY INSTRUMENTATION

A preventive maintenance program for the instrumentation ensures fewer interruptions of analyses, personnel efficiency, and lower repair costs. It eliminates premature replacement of parts, and reduces discrepancy among test results. It increases reliability of results.

The laboratory has established the following preventive maintenance program:

1. Each type of equipment/instrument has a written Standard Operating Procedure (SOP) which describes the methods for routine inspection, cleaning, maintenance, testing, calibration, and/or standardization of the equipment. Instrument operating manuals are kept near the instrument or where analysts have easy access.

2. Analysts using the instruments are properly trained and develop trouble-shooting skills in equipment failure to reduce dependence upon outside servicing agencies. In complicated cases, the servicing agency or supplier is called to solve the problem.

3. Written equipment records are kept to document all inspection, maintenance, trouble-shooting, calibration, or modifications. Whenever maintenance is performed on an instrument, it is properly documented in a preventive maintenance logbook, which is kept near the equipment to monitor the adequacy of maintenance schedules. The records contain the date (month, day, year), description of the maintenance done, the actual findings, the name of the person doing the maintenance and a statement of whether the maintenance operations were routine, and if those operations followed the written SOP.

4. Performance criteria is established for judging when data from instrument performance checks indicate the need to make adjustments in the instrument operating conditions. (See Section 4.0, Calibration Procedures and Frequency for details)

Chromatographic Instruments

Preventive maintenance is done through a daily performance check and calibration of standards. Parameters such as retention time and response factors are observed and back-checked with prior operational performance.

In addition, the following are done:

- GC detectors are cleaned whenever performance degradation (i.e., calibration criteria are not met, retention time shifts, noisy baseline, etc.) is observed by the analyst.
- Septa are replaced as needed.
- Incoming gas drying cartridges are changed whenever the color of the adsorbent is noticed.
- Effluent adsorbent traps are changed every month.
- Columns (GC and HPLC) are checked by performance and operating conditions when in use or prior to use.
- Oven performance checked daily prior to use.

(See Appendix D for SOP for GC Preventive Maintenance for more detailed discussion)

Gas Chromatography/Mass Spectroscopy (GC/MS)

The preventive maintenance includes:

Maintenance Requirements	Frequency
Clean filters on cooling fans	Monthly
Check cooling fan for proper operation	Monthly
Check line voltage	Monthly
Clean CDC disc drive pre-filter	Monthly
Check cool-flow level	Monthly
Change mechanical pump oil	Every 4 months
Clean source and rods	Every 4 months
Check power supplies in QEM Box	Every 4 months
Clean printer inside and outside	Every 4 months
General cleaning of instrument	Every 4 months
All items from monthly maintenance schedule	Every 4 months
Change primary filters on CDC disc drive	Every 6 months
Sensitivity analysis through BFB and DFTPP tune criteria	Every 12-hour clock

Atomic Absorption Spectrophotometers/ICP

Preventive maintenance is done for atomic absorption through the following checks:

- Minimum warm-up period of 30 minutes
- Alignment of hollow cathode tube to produce the maximum emitted light to the detector
- For flameless AA, the inert gas flow inside the furnace is optimized to ensure maximum sensitivity
- Digital readout values obtained for the standard curve of each element are checked to ensure linearity
- If readings are low, the operator checks the gas flows, burner or cell alignment, wavelength, slit width, photomultiplier voltage and lamp intensity prior to analysis

General Laboratory Equipment

Analytical balances of various capacities and operational modes are calibrated annually by a licensed specialist and officially recorded as verification of performance.

Balances are calibrated with standard Class S calibration weights before usage. The pH/specific ion meters are calibrated before use with a minimum of two standard solutions. All combination pH electrodes will be stored in pH 4 buffer solutions. Appendix D shows SOPs used for some instrumentation and equipment.

10.0 DATA ASSESSMENT PROCEDURES

10.1 FIELD DATA

Precision

Duplicate and replicate samples analyzed by the laboratory assess the precision of the sampling effort. Control limits for duplicate/replicate RPDs are set at 0-20% to provide interim guideline. Once a sufficient amount of replicate data becomes available, field precision control charts are constructed similar to the laboratory precision charts. For any given concentration, the mean and the standard deviation(s) of the replicates are calculated. The mean is the centerline of the control chart. Data from each sample set are pooled with the previous sample sets to generate control and warning limits for the next set. Warning and control limits for water samples are set at ±2s and ±3s, respectively. Control limits for solid samples are more liberally established due to matrix heterogeneity. Data outside any control limit are subject to QA review.

Accuracy

Field instruments are calibrated daily or more frequently, if needed, to ensure accuracy of the measurement of field parameters. Specifically, the pH measurement must be accurate to ±0.1 unit, temperature must be to ±0.5°C, and specific conductance must be ±10 umhos/cm. Each will be purged until the above parameters are stable within the specified limits.

All blanks associated with each sample set are to be analyzed and evaluated for cross-contamination. Blank contamination and the resulting corrective action are assessed on an individual basis.

Completeness

The Field Supervisor is responsible for ensuring that all field instrumentation and equipment is functioning properly and calibrated according to set procedures, and that all data are recorded accurately and legibly. In addition, the Field Supervisor must ensure all sites are sampled for all of the specified analyses, that sufficient sample volume has been provided to complete those analyses, and that all of the QA samples have been included with each sample set. The goal for completeness for each sample set shipped to the laboratory is 100 percent. The minimum acceptable completeness limit is 95 percent.

10.2 LABORATORY DATA

Accuracy

Data accuracy is a reflection of the efficiency of the analytical procedure. It is determined by use of spiked samples and standard reference materials or laboratory control samples performed at the rate of one set every 20 samples. A control chart is generated using historical laboratory data where warning and control limits are established to assess data accuracy. (See Appendix E for Laboratory Control Limits)

Precision

Precision is based upon the results of the relative percent differences as calculated from the percent recoveries of the matrix spike and duplicate samples. The control limits for precision are based on historical laboratory data. (See Appendix E for Laboratory Control Limits)

Completeness

Completeness is expressed as the percentage of the amount of valid data obtained to the amount of data expected. For a set of data to be considered complete, it must include all QC data verifying its accuracy and precision. (See Section 6.2, Laboratory Data, for details.)

Comparability

Comparison of results from one batch of samples to another is established by the analysis of the Laboratory Control Samples (LCS).

11.0 CORRECTIVE ACTION

11.1 FIELD CORRECTIVE ACTION

The initial responsibility for monitoring the quality of field measurements lies with the field personnel. The Field Supervisor is responsible for verifying that all QC procedures are followed. This requires that the Field Supervisor assess the correctness of the field methods, the ability to meet QA objectives, and to make a value judgement of the impact a procedure has upon the field objectives and subsequent data quality. If a problem occurs that might jeopardize the integrity of the project, cause a QA objective not to be met, or impact data quality, the Field Supervisor will immediately notify the PO and the Technical Operations Manager if appropriate. Corrective action measures are then decided upon and implemented. The PO is notified if the situation warrants it. The Field Supervisor documents the situation, the field objective affected, the corrective action taken, and the results of that action.

11.2 LABORATORY CORRECTIVE ACTION

Procedures for Determining and Reporting Out-of-Control Events

Out-of-Control Event:
An out-of-control event is defined as any occurrence failing to meet the QA/QC plan.

Criteria Used for Determination of an Out-of-Control Event:
Factors that affect data quality (failure to meet calibration criteria, inadequate record keeping, improper storage, or preservation of samples) require investigation and corrective actions.

Some factors can be easily assessed through the use of control chart interpretation. Control charts can reveal shifts, trends, biases, and conditions where parts of the analytical system are out-of-control. One or more of the data points in the control chart are out-of-control when:

- One or more data points fall outside the control limits (3s)
- A run of two or more data points outside the warning limits (±2s)
- A run of seven or more successive data points above x or
below x indicating trends or shifts
- A run of five successive data points in the same direction
- A cyclical pattern or non-random pattern appears in the control chart data

The detection of one of these conditions is an indication that the analytical system is out-of-control. The out-of-control value(s) is placed on the control chart and circled. The QA Officer is notified and both the analyst and QAO investigate and determine whether the condition indicates a procedure that is truly out-of-control or a possible random error. The QAO shall document corrective actions taken (i.e. whether the sample run was repeated or whether the data was received and released for reporting to the client) on the corrective action form.

Responding to an Out-of-Control Event

Roles and Responsibilities:
When an out-of-control event is recognized, each individual involved with the analysis in question has an interactive role and responsibility, these are as follows:

- **The Analyst:** He must be able to recognize QC failure and immediately notify the Laboratory Supervisor and work with the QAO to solve the problem.

- **The Laboratory Supervisor:** He must review all analytical and QC data for reasonableness, accuracy, and clerical errors; also responsible to monitor QC charts (in terms of control limits). In an out-of-control event, the laboratory manager works with the analyst and QAO to solve the problem and prevents the reporting of suspect data by stopping work on the analysis in question and ensuring that all results that are suspect are repeated, if possible, after the source of the error is determined and remedied.

- **QA Officer:** In the event an out-of-control situation occurs that is unnoticed at the bench or supervisory level (i.e., performance failure on a QC sample), the QAO will notify the laboratory manager, help identify and solve the problem where applicable, and ensure the work is stopped on the analysis and no suspect data is reported.

Procedures for Stopping Analysis

Whenever the analytical system is out-of-control, investigation/ correction efforts are initiated by all concerned personnel.

If the problem is instrumental or specific only to preparation of that sample batch, samples prepared after the out-of-control event are processed after the instrument is repaired and recalibrated, provided holding times are not exceeded.

If a sample batch is still out-of-control after reanalysis, all method-related activities shall stop immediately. A detailed laboratory-wide investigation shall be conducted to isolate and correct faulty operations. Sample security, integrity of standards, reagents, glassware, laboratory notebooks, instrument performance and adherence to the methods shall be included in the investigation.

All actions shall be documented and placed in their respective case/contract file.

Corrective Action

The need for corrective action comes from several sources: equipment malfunction; failure of internal QA/QC checks; failure of performance of system audits; and noncompliance with QA requirements.

When measurement equipment or analytical methods fail QA/QC, the problems will immediately be brought to the attention of the PO and QAO. Corrective measures to be taken will depend entirely on the type of analysis, the extent of the error, and whether the error is determinant or not. The corrective action to be taken is determined by either the Technical Operations Manager, the analyst, the PO or the QA officer or by all of them in conference.

A corrective action can be as extensive as replacing a complete lot of contaminated extraction solvent, the reextracting and analyzing of a complete batch of samples due to reagent blank contamination, or as simple as recalculating a series of results because a wrong dilution factor was applied. Furthermore, the right corrective action must be determined on a case by case basis. Appendix D outlines a list of potential analytical problems that might be encountered in the laboratory.

If failure is due to equipment malfunction, the equipment will be repaired, precision and accuracy will be reassessed, and the analysis will be rerun. All attempts will be made to reanalyze all affected parts of the analysis so that in the end, the product is not affected by failure of QA requirements.

When a result in a performance audit is unacceptable, the laboratory will identify the problems and implement corrective actions immediately. A step-by-step analysis and investigation to determine the cause of the problem shall take place as part of the corrective action program. If the problem cannot be controlled, the laboratory will analyze what the impact will be on the data results.

When a system audit reveals an unacceptable performance, work shall be suspended until corrective action has been implemented and performance has been proven to be acceptable.

All corrective actions will be documented on the Laboratory Corrective Action Report Form. These Reports are discussed weekly by the supervisors, managers, QA Officer, and Laboratory Director.

A follow-up review is then performed on each corrective action to ensure that the response taken has indeed been incorporated into the laboratory operation, minimizing a reoccurrence of the problem.

LABORATORY CORRECTIVE ACTION

➤ Contract/Project/Episode Number:

DESCRIPTION OF EVENT

➤ Date Recognized: ➤ By:

➤ Date Occurred: ➤ By:

➤ Date Corrected: ➤ By:

ANALYTICAL METHOD

➤ Analyte: ➤ Method:

➤ Analyst: ➤ Lab Manager:

➤ Samples Affected (Lab ID):

➤ Description of problem encountered:

➤ Corrective/Preventive Action Taken:

➤ **SIGNATURES:**

Analyst: *Date:*

Supervisor *Date:*

QA Officer *Date:*

FOLLOW UP

➤ Follow up Investigation/Discussion:

➤ **SIGNATURES:**

QA Officer: *Date:*

Laboratory Director: *Date:*

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FIG. 11.1 Laboratory Corrective Action Form

Remedial Actions

Remedial Actions are taken when a sample has to be reextracted due to poor surrogate recovery, improper level of extraction, etc. The Remedial Action Form is filled out by the analytical chemist and submitted to the section supervisor. The supervisor review the problem and signs the form, then sends it on to the extraction lab for the remedial action.

REMEDIAL ACTION RECORD

GC/MS DEPARTMENT

Initiated by:_____ **Project*:**_____

Date:_____ **Client*:**_____

Sample ID's:_____ **Case*:**_____

*if applicable

DESCRIPTION OF PROBLEM (list dates):

Supervisor's Signature:_____

DESCRIPTION OF ACTIONS TAKEN (list dates):

Supervisor's Signature:_____

Copies: **Case File,** _____

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FIG. 11.2 Remedial Action Record

12.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

12.1 QUALITY ASSURANCE REPORTS TO MANAGEMENT

Data from formal performance audits of the laboratory's activities are reviewed directly by the QAO, Laboratory Director, and Laboratory Manager.

Should any significant quality assurance problem arise, it will be internally discussed among the QAO, Laboratory Director, and Laboratory Manager at a weekly meeting.

13.0 REFERENCES

1. 40 CFR, Part 136, Federal Register, Volume 49, Number 209, Friday October 26, 1984.

2. Handbook for Analytical Quality Control in Water and Wastewater Laboratories, U.S. EPA 600/4-79-019, March 1979.

3. Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, USEPA, EPA-600/4-82-057, July 1982, and as subsequently amended.

4. "Principles of Environmental Analysis," Analytical Chemistry Volume 55, pp. 2210-2218, December 1983.

5. Quality Assurance Handbook of Air Pollution Measurement Systems, USEPA 600/9-76-005, December 1984.

6. Statement of Work: Multi-Media USEPA Contract Laboratory Program USEPA, 1987.

7. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, USEPA SW-846, 3rd Edition, September 1986.

8. USATHAMA QA Program, 2nd Edition, March 1987.

9. A Compendium of Superfund Field Operations Methods, USEPA, EPA/540/P-87/001, December 1987.

APPENDIX A

ANALYTICAL PROTOCOLS UTILIZED AT THE SWOK LABORATORY

1. Standard Methods for the Examination of Water and Wastewater: APHA, AWWA, and WPCG; 16th Edition, 1985,
2. Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act., USEPA.
3. 40 CFR, Part 136, Federal Register, Volume 49, Number 209, Friday, October 26, 1984.
4. Methods for Chemical Analysis of Water and Wastes, USEPA, EPA-600/4-79-020, March 1979 and as amended December, 1982 (EPA-600/482-055)
5. Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, USEPA, EPA-600/4-82-057, July 1982 and as subsequently amended.
6. Pesticide Analytical Manual, Volume I, Food and Drug Administration, Revised September, 1977.
7. Recommended Methods of Analysis for the Organic Components Required for AB1803, State of California, 5th Edition, April 1986.
8. NIOSH Manual of Analytical Methods, U.S. Department of Health and Human Services, 3rd Edition, February, 1984.
9. Statement of Work: Multi-Media USEPA Contract Laboratory Program, USEPA, 1990.
10. Statement of Work: Multi-Media High Concentration, USEPA Contract Laboratory Program, July 1986.
11. 500 Series Methods-Drinking Water Test Methods, USEPA Compendium of Methods.
12. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, USEPA, EPA-600/4-84-041, April 1984.
13. Annual Book of ASTM Standards, Water and Environmental Technology, ASTM, 1987, Volume 11.01 and 11.02
14. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, USEPA SW-846, 3rd Edition, September, 1986.

APPENDIX B

DEFINITION AND PROCEDURE FOR THE DETERMINATION OF THE METHOD DETECTION LIMIT

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero and determined from analysis of a sample in a given matrix containing analyte.

Scope and Application

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample.

The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device- or instru.. ent-independent.

Procedure

1. Make an estimate of the detection limit using one of the following:

   (a) The concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5. If the criteria for qualitative identification of the analyte is based upon pattern recognition techniques, the last abundant signal necessary to achieve identification must be considered in making the estimate.

   (b) The concentration value that corresponds to three times the standard deviation of replicate instrumental measurements for the analyte in reagent water.

   (c) The concentration value that corresponds to the region of the standard curve where there is a significant change in sensitivity at low analyte concentrations, i.e., a break in the slope of the standard curve.

   (d) The concentration value that corresponds to known instrumental limitations. It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the estimate of the detection limit.

2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferant concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferant). The interferant concentration is presupposed to be normally distributed in representative samples of a given matrix.

3. (a) If the MDL is to be determined in reagent water (blank), prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated MDL. (Recommend between 1 and 5 times the estimated MDL.) Proceed to Step 4.

   (b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated MDL proceed to Step 4.

   If the measured concentration of analyte is less than the estimated MDL, add a known

amount of analyte to bring the concentration of analyte to between one and five times the MDL in the case where an interference is co-analyzed with the analyte.

If the measured level of analyte is greater than five times the estimated MDL, there are two options:

(1) Obtain another sample of lower level of analyte in same matrix if possible.

(2) The sample may be used as is for determining the MDL if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.

4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the MDL and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If blank measurements are required to calculate the measured level of analyte, obtain separate blank measurements for each sample aliquot analyzed. The average blank measurements is subtracted from the respective sample measurements.

(b) It may be economically and technically desirable to evaluate the estimated MDL before proceeding with 4a. This will: (1) prevent repeating this entire procedure when the costs of analyses are high, and (2) ensure that the procedure is being conducted at the correct concentration. It is quite possible that an incorrect MDL can be calculated from data obtained at many times the real MDL even though the background concentration of analyte is less than five times the calculated MDL. To ensure that the estimate of the MDL is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower MDL. Take two aliquots of the sample to be used to calculate the MDL and process each through the entire method, including blank measurements as described above in 4a. Evaluate these data:

(1) If these measurements indicate the sample is in the desirable range for determining the MDL, take five additional aliquots and proceed. Use all seven measurements to calculate the MDL.

(2) If these measurements indicate the sample is not in the correct range, reestimate the MDL, obtain new sample as in 3 and repeat either 4a or 4b.

5. Calculate the variance (S^2) and standard deviation (S) of the replicate measurement as follows:

$$S2 = \left[\frac{1}{n-1} \left(\sum_{i=1}^{n} X_i^2 - \sum_{i=1}^{n} X_i \right) \Big/ {}^{2}n \right]$$

$$S = (S^2)^{1/2}$$

where the X_1, i = 1 to n are the analytical results in the final method reporting units obtained from the n sample aliquots and

$$\sum_{i=1}^{n} X_i^2$$

refers to the sum of the X values from i = 1 to n.

6. (a) Compute the MDL as follows:

$$MDL = t_{(n-1\ 1-o=99)} \quad (S)$$

where MDL = the method detection

$t_{(n-1\ 1-o=99)}$ =the analyst's "t" value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See Table.

S = standard deviation of the replicate analyses.

(b) The 95% confidence limits for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution (X^2/df) and calculated as follows:

$MDL_{LCL} = 0.69\ MDL$
$MDL_{UCL} = 1.92\ MDL$

where MDL_{LCL} and MDL_{UCL} are the lower and upper 95% confidence limits respectively based on seven aliquots.

7. Optional iterative procedure to verify the reasonableness of the estimated MDL and calculated MDL of subsequent MDL determinations.

   (a) If this is the initial attempt to compute MDL based on the estimated MDL in Step 1, take the MDL as calculated in Step 6, spike in the matrix at the calculated MDL and proceed through the procedure starting with Step 4.

   (b) If the current MDL determination is an iteration of the MDL procedure for which the spiking level does not permit qualitative identification, report the MDL as that concentration between the current spike level and the previous spike level which allows qualitative identification.

   (c) If the current MDL determination is an iteration of the MDL procedure and the spiking level allows qualitative identification, use S^2 from the previous MDL calculation to compute the F ratio.

$$\text{if} \quad \frac{S^2_A}{S^2_B} < 3.05$$

then compute the pooled standard deviation by the following equation:

$$S_{pooled} = \left[\frac{6S^2_A - 6S^2_B}{12} \right]^{1/2}$$

$$\text{if} \quad \frac{S^2_A}{S^2_B} < 3.05$$

re-spike at the last calculated MDL and proceSS the samples through the procedure starting with step 4.

   (c) Use the S_{pooled} as calculated in 7b to compute the final MDL according to the following equation:

$$MDL = 2.681\ (S_{pooled})$$

where 2.681 is equal to $t_{(n-1\ 1-o=99)}$ (S).

   (d) The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from percentiles of the chi squared over degrees of freedom distribution.

$$MDL_{LCL} = 0.72\ MDL$$
$$MDL_{UCL} = 1.65\ MDL$$

where LCL and UCL are the lower and upper 95% confidence limits respectively based on 14 aliquots.

Reporting

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units. If the analytical method permits options which affect the method detection limit, these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with MDL value. Report the mean analyte level with the MDL. If a laboratory standard or a sample that contained a known amount analyte was used for this determination, report the mean recovery, and indicate if the MDL determination was iterated.

If the level of the analyte in the sample matrix exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL.

Reference

Glaser, J.A., Foerst, D.L., McKee, G.C., Quave, S.A., and Budde, W.L., "Trace Analysis for Wastewaters", Environmental Science and Technology, 15, 1426 (1981)

TABLE B.1 ANALYST'S VALUES AT THE 99 PERCENT CONFIDENCE LEVEL

Number of Replicates	Degrees of Freedom (n-1)	$t_{[n-1\,1-\alpha=99]}$
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
		2.326

TABLE B.2 METHOD DETECTION LIMITS

PARAMETERS	METHOD REFERENCE	MDL	
		WATER (mg/L)	SEDIMENT (mg/Kg)
TOTAL PETROLEUM HYDROCARBONS	E418.1	0.011	.379
TOTAL DISSOLVED SOLIDS	E160.1	9.00	
CHLORIDE	E300.0	0.16	
FLUORIDE		0.07	
SULFATE		0.35	
NITRATE		0.07	
ORTHO-PHOSPHATE		0.45	
NITRATE	E353.2	0.01	
NITRITE	E354.1	0.02	
ARSENIC	SW7060	0.0015	.30
LEAD	SW7421	0.0007	.14
MERCURY	SW7470	0.0001	.02
SELENIUM	SW7740	0.0039	.78
THALLIUM	SW7841	0.0015	.30
ALUMINUM	SW6010	0.008	1.60
ANTIMONY		0.032	6.50
ARSENIC		0.021	4.20
BARIUM		0.007	1.40
BERYLLIUM		0.0002	.04
CADMIUM		0.002	.34
CALCIUM		0.246	49.00
CHROMIUM		0.003	.68
COBALT		0.003	.68
COPPER		0.013	2.60
IRON		0.003	.58
LEAD		0.041	8.20
MAGNESIUM		0.135	27.00
MANGANESE		0.0009	0.18
MOLYBDENUM		0.005	1.10
NICKEL		0.005	1.00
POTASSIUM		0.312	62.00
SELENIUM		0.060	12.00
SILVER		0.002	0.46
SODIUM		0.172	34.00
THALLIUM		0.021	4.20
VANADIUM		0.002	0.40
ZINC		0.002	0.44
CYANIDE	SW9012	0.0005	0.27

TABLE B.2 METHOD DETECTION LIMITS (continued)

PARAMETERS	METHOD REFERENCE	MDL	
		WATER (µg/L)	SEDIMENT (mg/Kg)
BENZENE	SW8020	.08	.0001
TOLUENE		.12	.0001
CHLOROBENZENE		.07	.0001
ETHYLBENZENE		.09	.0001
m,p-XYLENE		.08	.0001
o-XYLENE		.06	.0001
1,3-DICHLOROBENZENE		.05	.0001
1,4-DICHLOROBENZENE		.06	.0001
1,2-DICHLOROBENZENE		.05	.0001
CHLOROMETHANE	SW8010	.13	.0001
VINYL CHLORIDE		.11	.0001
BROMOMETHANE		.23	.0002
CHLOROETHANE		.15	.0001
TRICHLORO FLUOROMETHANE		.12	.0001
1,1-DICHLOROETHANE		.05	.0001
METHYLENE CHLORIDE		.10	.0001
trans-1,2-DICHLOROETHENE		.04	.0001
1,1-DICHLOROETHANE		.05	.0001
cis-1,2-DICHLOROETHENE		.05	.0001
CHLOROFORM		.08	.0001
1,1,1-TRICHLOROETHANE		.07	.0001
CARBON TETRACHLORIDE		.06	.0001
1,2-DICHLOROETHANE		.04	.0001
TRICHLOROETHENE		.06	.0001
1,2-DICHLOROPROPANE		.04	.0001
BROMODICHLOROMETHANE		.05	.0001
2-CHLOROETHYL VINYL ETHER		.35	.0004
cis-1,3-DICHLOROPROPENE		.03	.0001
trans-1,3-DICHLOROPROPENE		.03	.0001
TETRACHLOROETHENE		.08	.0001
DIBROMOCHLOROMETHANE		.04	.0001
CHLOROBENZENE		.05	.0001
BROMOFORM		.04	.0001
1,1,2,2-TETRACHLOROETHANE		.03	.0001
para-CHLOROTOLUENE		.37	.0004
1,3-DICHLOROBENZENE		.03	.0001
1,4-DICHLOROBENZENE		.07	.0001
1,2-DICHLOROBENZENE		.04	.0001

TABLE B.2 METHOD DETECTION LIMITS (continued)

PARAMETERS	METHOD REFERENCE	MDL	
		WATER (µg/L)	SEDIMENT (µg/Kg)
NDMA	SW8280M	0.05	
DICHLORVOS	SW8140	0.096	3.17
MEVINPHO		0.277	9.14
ETHOPROP		0.165	5.45
NALED		0.106	3.50
PHORATE		0.099	3.27
DEMETON-S		0.227	7.49
DIAZINON		0.397	13.10
DISULFOTON		0.142	4.69
METHYL PARATHOIN		0.014	0.46
RONNEL		0.181	5.97
MALATHION		0.050	1.65
FENTHION		0.079	2.61
CHLORPYRIFOS		0.223	7.36
STIROPHOS		1.28	42.20
MERPHOS		0.249	8.22
FENSULFOTHION		2.29	75.60
BOLSTAR		0.137	4.52
AZINPHOS METHYL		1.16	38.30
COUMAPHOS		1.07	35.30

PARAMETERS	METHOD REFERENCE	MDL	
		WATER (µg/L)	SEDIMENT (mg/Kg)
ALPHA-BHC	SW8080	.004	.00027
BETA-BHC		.004	.00027
DELTA-BHC		.004	.00017
GAMMA-BHC(LINDANE)		.003	.00029
HEPTACHLOR		.008	.00102
ALDRIN		.004	.00029
HEPTACHLOR EPOXIDE		.003	.00034
ENDOSULFAN I		.004	.00030
DIELDRIN		.006	.00049
4,4-DDE		.007	.00049
ENDRIN		.009	.00053
ENDOSULFAN II		.007	.00066
4,4-DDT		.009	.00052
ENDRIN KETONE		.008	.00066
ENDRIN ALDEHYDE		.009	.00046
METHOXYCHLOR		.034	.00318
ALPHA-CHLORDANE		.004	.00031
GAMMA-CHLORDANE		.004	.00036
TOXAPHENE		.069	.0051
AROCHLOR-1016		.070	.0044
AROCHLOR-1221		.072	.0060
AROCHLOR-1232		.146	.0034
AROCHLOR-1242		.089	.005
AROCHLOR-1248		.067	.0028
AROCHLOR-1254		.038	.0048
AROCHLOR-1260		.096	.0034

TABLE B.2 METHOD DETECTION LIMITS (continued)

PARAMETERS	METHOD REFERENCE	MDL	
		WATER (µg/L)	SEDIMENT (µg/Kg)
ETHYLENE DIBROMIDE	E504.1	.005	.400
NAPHTHALENE	SW8310	.90	60.30
ACENAPHTHYLENE		.20	13.40
ACENAPHTHENE		.90	60.30
FLUORENE		.10	6.70
PHENANTHRENE		.04	2.68
ANTHRACENE		.01	0.67
FLUORANTHENE		.02	1.34
PYRENE		.07	4.69
BENZO(A)ANTHRACENE		.02	1.34
CHRYSENE		.06	4.02
BENZO(b)FLUORANTHENE		.02	1.34
BENZO(k)FLUORANTHENE		.01	0.67
BENZO(a)PYRENE		.01	0.67
DIBENZO(a,h)ANTHRACENE		.20	13.40
BENZO(g,h,i)PERLYENE		.20	13.40
INDENO(1,2,3-cd)PYRENE		.10	6.70
DALAPON	SW8150	.182	2.33
DICAMBA		.077	1.79
MCPP		65.900	1173.00
MCPA		54.300	1034.00
DICHLOROPROP		.142	3.08
2,4-D		.104	2.43
2,4,5-TP		.051	1.28
2,4,5-T		.046	1.05
DINOSEB		.031	0.860
2,4-DB		.358	6.37
HMX	SW8330	7.55	57.5
RDX		6.66	63.1
TNB		5.66	67.6
TETRYL		8.06	436.0
DNB		1.63	76.9
TNT		2.17	59.3
NITROBENZENE		2.27	71.5
26DNT		6.10	52.8
24DNT		3.81	42.1
2NT		7.58	80.3
4NT		14.30	51.7
3NT		7.90	17.2

TABLE B.2 METHOD DETECTION LIMITS (continued)

PARAMETERS	METHOD REFERENCE	MDL	
		WATER (µg/L)	SEDIMENT (µg/Kg)
	SW8270		
Acenaphthene		4.22	1.39
Acenaphthylene		4.14	1.64
Anthracene		1.43	2.94
Benzo(A)Anthracene		2.89	1.68
Benzo(B)Fluoranthene		7.14	3.21
Benzo(K)Fluoranthene		15.21	2.24
Benzo(G,H,I)Perylene		7.76	4.72
Benzo(A)Pyrene		2.05	1.73
Benzyl Alcohol		14.05	9.03
Bis(2-Chloroethoxy)Methan		3.98	2.14
Bis(2-Chloroethyl)ether		5.14	2.24
Bis(2-chloroisopropyl)ether		9.93	2.65
Bis(2-Ethylhexyl)Phthalate		4.71	5.10
4-Bromophenylphenylether		1.30	1.17
Butylbenzylphthalate		2.30	2.59
4-Chloroanaline		21.14	8.58
2-Chloronaphthalene		9.44	1.55
4-Chlorophenyl-Phenylethe		2.80	1.85
Chrysene		6.94	2.29
Dibenz(A,H)Anthracene		6.92	4.77
Dibenzofuran		3.14	1.81
Di-N-Butylphthalate		4.02	1.94
1,2-Dichlorobenzene		5.75	1.47
1,3-Dichlorobenzene		4.71	2.22
1,4-Dichlorobenzene		4.58	2.00
3,3'-Dichlorobenzidine		22.66	21.13
Diethylphthalate		3.58	1.36
Dimethylphthalate		6.40	0.96
2,4-Dinitrotoluene		6.28	2.96
2,6-Dinitrotoluene		3.35	2.09
Di-N-Octylphthalate		5.78	11.33
Fluoranthene		6.28	4.38
Fluorene		1.91	1.31
Hexachlorobenzene		2.00	1.98
Hexachlorobutadiene		11.43	2.95
Hexachlorocyclopentadiene		0.00	3.79
Hexachloroethane		4.51	2.45
Indeno(1,2,3-CD)Pyrene		6.33	9.63
Isophorone		2.33	1.79
2-Methylnaphthalene		5.44	2.34
Naphthalene		16.24	2.65
2-Nitroaniline		8.01	6.08
3-Nitroaniline		7.64	22.06
4-Nitroaniline		33.47	38.70
Nitrobenzene		4.25	1.42
N-Nitrosodiphenylamine		2.38	2.46
N-Nitroso-di-n-propylamine		5.24	2.88
Phenanthrene		3.42	1.90

TABLE B.2 METHOD DETECTION LIMITS (continued)

PARAMETERS	METHOD REFERENCE	MDL	
		WATER (µg/L)	SEDIMENT (µg/Kg)
	SW8270 (cont.)		
Pyrene		4.07	2.61
1,2,4-Trichlorobenzene		15.40	2.76
Benzonic Acid		47.61	2.88
4-Chloro-3-Methylphenol		2.58	3.60
2-Chlorophenol		2.88	1.49
2,4-Dichlorophenol		2.11	4.36
2,4-Dimethylphenol		7.35	2.31
4,6-Dinitro-2-Methylphenol		19.93	14.81
2,4-Dinitrophenol		15.16	29.81
2-Methylphenol		4.60	1.60
4-Methylphenol		4.60	1.87
2-Nitrophenol		2.92	3.41
4-Nitrophenol		20.05	14.91
Pentachlorophenol		7.23	14.60
Phenol		6.97	3.16
2,4,5-Trichlorophenol		3.66	1.27
2,4,6-Trichlorophenol		3.77	2.35
2-Fluorophenol		12.39	4.51
2-Fluorobiphenyl		4.89	2.55
2,4,6-Tribromophenol		10.65	6.50
Carbazole		2.58	3.35
Aniline		0.36	1.46
1-Methylnaphthalene		0.54	0.43

TABLE B.2 METHOD DETECTION LIMITS (continued)

PARAMETERS	METHOD REFERENCE	MDL	
		WATER (µg/L)	SEDIMENT (µg/Kg)
	SW8240		
Acetone		3.18	7.03
Benzene		0.67	0.23
Bromodichloromethane		0.57	0.11
Bromoform		0.78	0.59
Bromomethane		1.20	0.16
2-Butanone (MEK)		6.38	2.15
Carbon Disulfide		0.59	0.31
Carbon Tetrachloride		1.56	0.24
Chlorobenzene		0.71	0.20
Chloroethane		0.89	0.95
2-Chloroethyl vinyl ether		2.95	-
Chloroform		0.46	0.16
Chloromethane		1.17	0.41
Dibromochloromethane		0.81	0.19
1,1-Dichloroethane		0.64	0.22
1,2-Dichloroethane		0.98	1.17
1,1-Dichloroethene		0.86	0.17
cis-1,2-Dichloroethene		0.87	0.14
trans-1,2-Dichloroethene		0.67	0.17
1,2-Dichloropropane		0.76	0.28
cis-1,3-Dichloropropene		0.74	0.31
trans-1,3-Dichloropropene		0.51	0.83
Ethylbenzene		0.75	0.33
Freon 113		0.56	0.64
2-Hexanone		1.75	2.34
Methylene Chloride		0.60	0.24
4-Methyl-2-Pentanone (MIBK)		2.25	1.85
Styrene		0.65	0.25
1,1,2,2-Tetrachloroethane		2.00	0.73
Tetrachloroethene		1.72	0.40
Toluene		0.91	0.24
1,1,1-Trichloroethane		1.27	0.24
1,1,2-Trichloroethane		1.43	1.24
Trichloroethene		1.37	0.22
Vinyl Acetate		0.92	2.15
Vinyl Chloride		0.91	0.33
o-Xylene		0.74	0.29
M,P,Xylene		1.68	2.15
1,3 Dichlorobenzene		1.65	0.34
1,4 Dichlorobenzene		1.63	0.33
1,2 Dichlorobenzene		1.61	0.37

APPENDIX C
Instrument Operations Conditions

INDUCTIVELY COUPLED ARGON PLASMA (ICAP) OPERATING CONDITIONS

Start Up:

1. Check that the argon tank main valve is open and the pressure is set to at least 60 psi.
2. Check the plasma work coil coolant water and exhaust vents.
3. Check that the drain tube is inserted into a plastic water bottle containing at least 8 inches of water and drain line is below surface.
4. On the R.F. generator control panel, turn the forward power manual control rheostat knob counter clockwise to the off position.
5. Check that the white power lamp is illuminated on the RF generator.
6. Turn the torch gas toggle switch on and adjust flow meter to 18, turn the auxiliary flow on to 0.5 and the sample gas flow to 0.6.
7. Purge the torch surfaces, the capillary tube and teh drainage tube of air for at least 3 minutes.
8. Check that the blue R.F. off lamp on the generator is illuminated.
9. Turn the Automatic Power Control switch to the manual position.
10. Press the red R.F. on button.
11. Turn the sample gas flow toggle switch to OFF position.
12. To ignite, slowly turn the forward power manual control rheostat knob clockwise until the forward power meter reads 0.5 KW.
13. Press the ignitor button on the front panel of the generator. You should see a faint filamentary plasma swirling in the outer tube of the torch. Gradually increase the forward power until the plasma ignites.
14. Once the plasma is lit, rotate the forward power rheostat knob until the forward power meter reading is 1.0 - 1.1 KW.
15. Turn the automatic power control switch to the automatic position.
16. Introduce the rinse solution into the plasma by slowly turning on toggle switch.
17. Turn off auxiliary flow toggle switch.

GAS CHROMATOGRAPH (GC) OPERATING CONDITIONS

The following are the gas chromatograph analytical conditions. The conditions are recommended unless otherwise noted.

Carrier Gas:	Helium (Hydrogen may be used)
Column Flow:	5mL/min.
Make-up Gas:	P-5/P-10 or N_2 (required)
Injector Temperature	$\geq$ 200°C
Injection:	On-column
Injection Volume:	1 or 2 μL
Injector:	Grob-type, splitless
Initial Temperature:	150°C
Initial Hold Time:	1/2 min.
Temperature Ramp:	5°C to 6°C/min.
Final Temperature:	275°C
Final Hold Time:	Until after Decachlorobiphenyl has eluted (approximately 10 minutes)

Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples, blanks, and MS/MSD's.

GC/MS SEMIVOLATILE OPERATING CONDITIONS

The following instrumental parameters are required for all performance tests and for all sample analyses:

Electron Energy: .....70 volts (nominal)
Mass Range:.....35 to 500 amu
Scan Time:.....1 second per scan
Initial Column
  Temperature Hold: .....40°C for 1 minute
Column Temperature
  Program:.....40-300°C at 8°/min.
Final Column
  Temperature Hold: .....300°C for 5 minutes
Injector Temperature:.....280°C
Transfer Line
  Temperature: .....380°C
Source Temperature: .....According to manufacturer's specifications
Injector: .....Grob-type, splitless
Sample Volume:.....2 μL

VOLATILE GC/MS OPERATING CONDITIONS

These performance tests require the following instrumental parameters:

Electron Energy:	70 Volts (nominal)
Mass Range:	35-300
Scan Time:	to give at least 5 scans per peak, not to exceed one second per scan.
Column conditions:	DB-624, 75m, 0.53mm ID
Film Thickness:	3 um
Flow Rate:	30 mL/min. (glass jets) 10-15mL min. (microjets)
Column Temperature:	Isothermal at 45°C for 5 min., then programmed at 5°C/min. to 105°C. Further programmed at 10°C/min. to 180°C

Adjust the purge gas (helium) flow rate to 35 ± 3 mL/min.

TABLE 1.
Instrument Operating parameters and Standard Conditions for Metals Analyzed by Graphite Furnace Atomic Absorption Spectrophotometry

Element	Wave Length (nm)	Slit Opening (mm)	HCL Lamp Current (mA)	EDL Lamp Watts (w)	Gas Setting Time/Rate	Furnace Conditions Dry [5]C/ Seconds	Char [6]C/ Seconds	Atomiz [6]C/ Sec.(ppm)
Arsenic	193.7	4 (1.0)	—	6.5	Ar(Normal/3 sec./20	110/22	400/22	2700/7
Lead	283.3	4 (1.0)	10	—	Ar(Normal/3 sec./20)	110/22	750/22	2300/7
Selenium	196.0	—	—	6.5	Ar(Normal/3 sec./20)	110/22	1200/22	2700/7
Thallium	276.8	4 (1.0)	20	—	Ar(Normal/3 sec./20)	110/22	400/22	2300/7

Element	Injection Volume (ul)	Linear Working Range(ppb	Sensitivity ppb/ .0044 Abs	Maximum Scale Expansion	Additional Elements
Arsenic	20	4-300	5	5	O[2] Correction Ni $(NO_3)_2$ matrix
Lead	20	0.3-100	2	30	O[2] Correction
Selenium	20	4-300	5	5	O[2] Correction Ni $(NO_3)_2$ matrix
Thallium	20	0.5-100	3	20	O[2] Correction

APPENDIX D
Related Standard Operating Procedures

LABORATORY AND SAMPLE SECURITY

Samples received at SWLO are considered to be physical evidence and are handled according to procedural safeguards established by the EPA. Because of the legal nature of the work, the laboratory provides complete security for samples, analysis and data.

To assure complete security for samples and analytical procedures during sample analyses, the following steps are taken:

- Sensitive Materials Containment Laboratory/ Process and Instrument Laboratories be locked at all times except when in actual use.
- Analytical sample should always be in the custody of an individual technician assigned to do the task.

The following security measures are employed:

- Doors to the laboratory are closed and secured at all times.
- Only authorized personnel and visitors under escort shall have access to the chemistry lab.
- All laboratory personnel should be aware of the need to question and determine legitimacy of a stranger's presence in the laboratory.
- Deliveries are to be escorted to the laboratory from the main reception area or from the receiving area.

TRACEABILITY OF STANDARDS

All inorganic and organic analytical standards utilized for instrument/methodological calibration and preparation of quality control samples shall be traceable to standard reference materials. Primary standards must be obtained from reliable certifiable source and be of the highest possible purity. Standards are purchased from approved commercial vendors such as Chem Services, Inc., Fisher Scientific, Supelco, etc., for the use in all laboratory analyses. Commercial standards prepared are verified against certified standard reference materials (SRM). Within the USEPA, the three sources of SRM that will be considered acceptable for traceability are:

a. Quality Assurance Materials Bank (QAMB)
b. Pesticides and Industrial Chemical Repository
c. Toxic and Hazardous Materials Repository

Whenever possible, SRM received from one of the three EPA repositories will be utilized directly for generation of control samples and analytical standards. However, if such reference materials are unavailable for routine use, standards will be definitively traceable to such reference materials.

For inorganic analytes, calibration standards and control samples shall be traceable to a standard reference material supplied by the National Bureau of Standards (NBS) or the USEPA's Toxic and Hazardous Materials Repository.

All standards must be stored under conditions and in containers that provide the greatest protection against deterioration and/or contamination.

Stock and working standards solutions must be made fresh as often as required by their stability and must be checked regularly for signs of deterioration (i.e., discoloration, formation of precipitates and changes in concentration). Standard solutions are properly labeled as to compound name, concentration, solvent, date and preparer.

(See Criteria and Guidelines of Analytical Standards and QC Samples for the Evaluation of Traceability.)

STANDARD EXPIRATION PROCEDURE

DEFINITIONS:

1. *Primary Compound* - A liquid or solid compound in a pure form obtained from an

approved commercial distributor. (It could have a certain % purity that can be used to correct standard concentration.)

2. *Stock Standard* - Standard prepared/or obtained directly from the primary compound (i.e., liquid or solid) at a high concentration. (Most commercially-prepared standards are convenient to be used as stock solutions.)
3. *Working Standards* - Standards used in the calibration and quantitation of the compounds of interest.

Expiration or Holding Time Criteria:

All standards obtained/or purchased from approved commercial vendors such as Chem Services, Inc., Fisher, Supelco, etc., as well as standard reference materials procured from EPA or NBS are dated upon receipt. Date of expiration is also noted and if not available will be obtained from the supplier or manufacturer; if no information is available from the supplier, the lab holding time or shelf-life for the materials obtained shall be half the normal shelf-life (i.e., assuming 1 year for most compounds, then it would be 6 months shelf-life).

Standards prepared as stock/or working standards are properly labeled as to name of compound mixture, concentration, solvent/medium, date and preparer.

Laboratory control sample (LCS) or an independent check standard available from EPA or NBS or standard reference materials (SRM) are used to monitor standard degradation. Check results of the LCS or the check standard are checked against the given target range values (i.e., it should be within the 95% confidence limits of the given values).

All working standards prepared from stock solution commercially obtained or prepared in-house must be checked against LCS or NBS or any certified reference material. Check results must be within the target range values (i.e., 95% confidence limits of the given values) of the LCS, NBS, or SRM or within 95% confidence limits of the true value of the check standard as determined from replicate analyses (assuming normal instrument sensitivity) previously determined. (See Criteria and Guidelines for the Preparation of Analytical Standards and Quality Samples.)

All standards used in the laboratory will have the following expiration or holding time criteria:

INORGANIC:

a. Stock Standards: Expiration date of commercially obtained standards is checked weekly to identify and purge outdated stock solutions

b. Working Standards: Standards are prepared at the following frequency depending on standard concentration:

   1. 1-5 ppm: remake every three weeks
   2. 0.1-1 ppm: remake every two weeks
   3. less than 0.1 ppm: remake every week

   Note: These standards can be prepared following the frequency stated above or sooner if comparison with a check standard indicates a problem. All working standards are evaluated by a check standard or LCS on a daily basis.

ORGANIC:

a. For liquid or solid stock standard:

Maximum holding time of 1 year from date of receipt if no expiration date is indicated by the manufacturer.

b. For commercially obtained prepared stock standards:

Prepare or obtain once a month or sooner if comparison with a check standard of LCS indicates a problem. (This would be based from the check results of the working standards prepared from this stock against the LCS or check standard.)

c. For working standards:

Prepare fresh weekly or sooner if comparison with a check standard or LCS indicates a problem. (This would be based from the check results of the working standards prepared from this stock against the LCS or check standard.)

CRITERIA AND GUIDELINES FOR THE PREPARATION OF ANALYTICAL STANDARDS AND QUALITY CONTROL SAMPLES

All standards obtained or purchased from approved commercial vendors shall be evaluated for traceability using standard reference materials (SRM) obtained from NBS and/or EPA reference materials.

A. CRITERIA FOR TRACEABILITY

Traceability to certified SRM shall be performed through statistical evaluation of the control sample or analytical standard relative to NBS and/or EPA reference material.

Criteria set for traceability shall be as follows:

1. The new standard solution/calibration standard is considered to be acceptable for sample quantification if the RPD (relative percent difference) is less than or equal to 10%.

   The general procedure used for this evaluation involves:

   a. Triplicate analysis of the certified SRM.

   b. Triplicate analysis of the newly prepared analytical standard or spiking solution within the same time frame.

   c. Mean and standard deviation statistics are calculated on each set of triplicate analyses.

   d. Relative Standard Deviation (RSD) of less than 10% must be obtained on each set of triplicate analyses for acceptability.

   e. The two sets of data (i.e., newly prepared standard or calibration standard solution) is then compared with the SRM to determine acceptability using criteria mentioned.

$$RPD = \frac{\text{Mean SRM value - Mean proposed standard value}}{\text{average of the Mean value}}$$

2. If SRM are not available, laboratory control samples (LCS) obtained from EPA shall be used for traceability. Working standards prepared from the stock solution (i.e., newly prepared standard) are used as calibration standards for the quantification of the LCS. Check results must be within the target range values (i.e., 95% confidence limits of true values) provided with the LCS.

3. If SRM and EPA LCS are not available, check results of the newly prepared standard must be within 95% confidence limits of the historical values obtained from replicate analysis of the same standard concentration previously determined (assuming normal instrument sensitivity).

B. GUIDELINES

The laboratory has established guidelines for the preparation of analytical standards used as spiking solutions and/or calibration standards which are as follows:

1. Laboratory technicians experienced in calibration and use of analytical measurement tools are assigned to do standard preparation tasks.

2. Analytical reagent grade materials, in solution or neat form, are utilized in the preparation of analytical/control sample standards. Whenever possible, guaranteed assay materials with supporting chromatograms are requested from the manufacturers.

3. Solvents used for dilution of standards are checked for background contamination. Contamination for dilution solvents is required in all phases of standard preparation. (See Solvent Check SOP)

4. Analytical measuring tools such as balance, volumetric glasswares, syringe, etc., are calibrated to obtain accurate measurements. (See Laboratory Equipment SOP)

5. All data generated (i.e., weights of standard used, volume aliquot taken, lot number, solvent used, date of preparation, concentration of the solution, etc.) are documented immediately in a standard preparation logbook.

6. A sequential standard log number (SL#) is assigned to the newly prepared standard solution. This standard identification code must be noted in the standard log, on all chromatograms generated from the instrument analysis of the solution for traceability evaluation, and on any storage uses which are used to contain the original

solution or any aliquots of the solution prepared.

7. Standards are analyzed prior to use for any analytical measurement by use of detection (i.e., GC detectors-FID, EC, and GC/MS) system it was intended for.
8. A standard of the same material obtained from NBS or EPA is used as quality control traceability reference standard. (See Criteria)
9. Both the new standard solution and the reference standard are analyzed on the same instrument and within the same time frame to maximize analytical precision.
10. The new standard solution is quantified against the reference standard as an unknown to determine its acceptability.
11. Once the standard solution has passed QC evaluation for traceability, it is aliquoted appropriately, flame sealed and stored at -4°C to maintain its integrity until required.

LIST OF EPA AND NBS REFERENCE STANDARDS

PARAMETERS		REFERENCE STANDARD
Inorganics		
Trace Metals	EPA:	ICAP QC Water Pollution QC Water Supply QC
	NBS:	SRM "3100" Series Spectrometric Solutions; SRM 1646 Estuarine Sediment SRM 1577a Bovine Liver
Organics		
Total Inorganic/ Organic Carbon	NBS:	SRM 191a, 192a, 84j
Volatile Organics	EPA:	Water Pollution QC Water Supply VOC I, II, IV,V, VI & VII Water Supply Trihalomethanes
Base/Neutrals/Acids	EPA:	Water Pollution QC GC/MS Acids Water Pollution QC GC/MS Base Neutral I, II, III
Pesticides	EPA:	Water Pollution QC GC/MS Pesticide I Water Pollution Chlorinated HC Pesticide I,II, III Water Supply Chlorinated HC Pesticide I, II
PCBs	EPA:	Water Pollution QC PCB 1016, 1260, 1248, 1254
EP Toxicity	EPA:	Water Pollution QC EP Extracts for Pesticides and Herbicides
Herbicides	EPA:	Water Supply QC

NOTE: Analytical reference standards for most organic parameters are obtained from EPA Quality Assurance Materials Bank, Pesticides and Industrial Chemical Repository and Toxic and Hazardous Materials Repository.

PREPARATION AND QA/QC PROCEDURE FOR THE LABORATORY WATER SUPPLY

The quality control of water used as reagent water or laboratory water involves consideration and control of the many variables that affect the production of reliable data. It should be free from interferences and other contaminants. Failure to prepare water properly and to use water suitably may account for the poor performance of some analytical methods.

The laboratory employs a high-purity ion-exchange system through the use of Millipore system that produces an ASTM Type II grade water. It consists of disposable cartridges for pre-filtration, organic absorption, deionization and Millipore filtration. The MilliQ water generated is checked for total dissolved solids not to exceed 0.1 mg/L. A new deionizing and filtration cartridge will be installed if purity falls below 12 megohms/cm.

The following preparation of laboratory water is employed for the different analyses done in the laboratory:

1. For trace metals and other general chemistry analyses

Deionized water generated by the ion-exchange system is used for trace elements and other inorganic work.

The presence of inorganic analytes is checked when the prepared reagent water is analyzed as reagent blank and/or calibration blank. For calibration blank analysis, any analyte concentration found should be less than the method detection limit (MDL) or required contract detection limit (CRDL). For preparation of blank analysis, if any analyte concentration in the blank is above the MDL or CRDL, the lowest concentration of that analyte in the associated samples must be 10X the blank concentration.

2. For volatile analysis

Reagent water is prepared by filtering deionized water through a carbon filter.

The presence of organic volatiles is chedked when the prepared reagent water is run as a method blank and/or system blank. When organic volatiles found (i.e., common laboratory solvents - methylene chloride, acetone and toluene), it should be <5x detection limit and no analytes in the Hazardous Substances List detected.

3. For organic extractions

Reagent water used for sample preparations as well as method blanks is preextracted bottled water or ASTM Type II water. The water is preextracted with a solvent of choice, i.e., depending on the method of extraction to be used. Most of the time the solvent used is methylene chloride or hexane (nanograde). The water is then used for method blank preparation as well as sample preparation for a given extraction method.

The presence of organic semivolatiles/pesticides is checked when the prepared reagent water is analyzed as a method blank. When organic semivolatiles/pesticides are found (i.e., common phthalate esters), there should be <5x detection limit and no analytes in the Hazardous Substances List detected.

To determine whether organic impurities are present in the bottled water, the solvent used for the pre-extraction is then concentrated to a suitable volume (~0.5 mL) and analyzed by chromatographic techniques, GC-FID or GC-ECD for the presence of organic contaminants. If peaks present in the GC-FID chromatogram are greater than 10% full scale deflection (FSD), the polished water is again re-extracted with the solvent of choice and reanalyzed for organic impurities.

NOTE: All calibration blank, system blank, preparation or method blank data are to be kept together with the associated samples analyzed.

STANDARD OPERATING PROCEDURE FOR CHECKING REAGENTS, SOLVENTS, AND GASES

A. Scope

Chemical reagents aside from the primary standard reagents, solvents, and gases are carefully selected to conform to specifications defined in the method of analyses. Selection is based on the required priority for parameters being measured, sensitivity of the method, and specificity of the detection system (i.e., AA, ICAP, GC-ECD, GC/MS).

B. Reagents

Laboratory reagents obtained from approved commercial vendors shall meet ACS standards and are labeled indicating contents, date of receipt or preparation, and expiration. Hazardous reagents are adequately labeled and stored segregated from the rest of the reagents to indicate type and degree of hazard.

C. Solvents

Solvent quality is also routinely monitored and checked for contamination prior to use by concentration (volume reduction) and GC analysis. All solvents are distilled-in-glass reagents which are purchased in large single lots (20-100 cases) and stored to enhance consistency. Solvent quality is checked each time a new lot is purchased and prior to use on all significant contracts. The following procedure is routinely employed to assess solvent quality:

1. A suitable volume of solvent (500mL) is accurately measured and placed in a 500mL KD flask with a 4mL receiver.
2. The solvent is reduced in volume using standard KD concentration techniques to a final volume which will produce a concentration factor of 100x (200uL) for a 500mL initial volume.
3. The concentrated solvent is then analyzed by both GC/ECD and GC/FID to evaluate the degree of contamination.
4. Any solvent producing extraneous peaks subject to interference with components of interest is discarded and replaced with solvent of adequate quality.
5. Solvent check chromatograms of the solvent lot number are kept in a file by the QA Officer for documentation.
6. Each lot of solvent is recorded in a solvent check logbook along with the date received, date of concentration, the analyst, and the results of the check (whether or not the lot has been approved.

D. Gases

Gases used in inorganic and organic analyses are of commercial grade or are laboratory supplied gases. For organic analyses, the type of detection (i.e., GC/ECD, Hall, GC/FID, GC/MS) used affects gas quality requirement. Molecular sieves, carrier-gas filters, and drying tubes are required on combustion gases to improve quality. Gas cylinders are immediately replaced when the pressure falls to 100-200 pounds per square inch (psi) to minimize detector contamination that will affect sensitivity of the detector.

STANDARD OPERATING PROCEDURE FOR GLASSWARE AND LABWARE CLEANING

To ensure the integrity of the samples, steps must be taken to minimize contamination from the containers the are stored in and through the glasswares or labwares used during sample analysis. If the analyte(s) to be determined is organic in nature, the container is preferably made of glass. If the analyte(s) is inorganic, then the container should be plastic or polyethylene.

When both organic and inorganic substances are to be analyzed, the following procedures should be taken when cleaning glassware or labwares that are to be used for sample analysis:

A. Metals Analysis Labware Washing Procedures

1. Wear safety glasses and polyethylene gloves.
2. Rinse with tap water to remove sample residue or reagents.
3. Wash with warm tap water and prepared "Microsoap".
4. Rinse with tap water followed by deionized water.
5. Soak labware in 3N HNO_3. (Be sure to thoroughly wet the entire inside.)
6. Rinse with tap water.
7. Soak labware in (deionized) water.
8. Allow labware to dry, covered with lab wipers at ambient laboratory temperature.
9. Store each labware in polyethylene bags and place in assigned storage area.

   NOTE: If a labware has been in storage for at least 2 weeks, the following procedures are taken before usage:

   a. Rinse labware in 3N HNO_3.
   b. Rinse at least twice with (deionized) water.
   c. Repeat the above steps 8-9.

B. Bottles for Hg Analysis: Cleaning Procedures.

1. Rinse with tap water.
2. Wash with prepared "Microsoap" and warm tap water.
3. Rinse with tap water.
4. Rinse with 20% $SnCl_2$ solution (made in 4N HCl).
5. Rinse with tap water followed by deionized water.
6. Soak in 3N HNO_3.
7. Rinse with deionized water.
8. Soak in (deionized) water for.
9. Rinse with (deionized) water after soaking.
10. Allow bottles to dry covered with lab wipers.
11. Put caps on bottles and place in polyethylene bags for storage in assigned areas.

NOTE: 3N HNO_3 soaking solution should be changed once a month.

C. Organic Analysis Glassware Preparation

Although procedures for glassware vary somewhat according to the type of apparatus involved, the protocol for most apparatus involves thorough washing, solvent rinsing and high temperature oxidation. This procedure has been proven extremely effective for elimination of contamination in trace level organic analyses. The glassware preparation procedure for non-volumetric glassware is as follows:

1. Rinse 1-2x with acetone or methanol to help remove residue.
2. Thoroughly wash in deionized water (DI) and detergent (Alconox) to remove particulate matter and gross contamination.
3. Rinse extensively with DI water.
4. Air dry.
5. Cover open ends and exposed portion of ground glass joints with aluminum foil and place in kiln at 200° C for 2 hours.

The following procedure is utilized for preparation of volumetric glassware:

1. Rinse 1-2x with acetone or methanol to help remove residue.
2. Thoroughly wash in DI water and Alconox to remove particulate matter and gross contamination.
3. Rinse extensively with DI water.
4. Submerge in concentrated "Nochromic"/ sulfuric acid bath for 2-4 hours and then rinse 5x with DI water (optional).
5. Rinse 2x with acetone or methanol.
6. Oven drying.
7. Cap with aluminum foil.
8. Rinse 3x with solvent of choice before using.

D. Segregation of Potentially Contaminated Glassware

When a sample is suspected of containing a high contaminant level, disposable glassware will be used as much as possible and discarded after use. Any non-disposable glassware that is used will be washed and prepared separately using the SOP for glassware preparation.

Glassware suspected of gross contamination will be rinsed with methylene chloride three times, the rinsate concentrated to a final volume of 0.5 mL, and analyzed by chromatographic techniques to check for residual contamination. Glassware checks exhibiting detectable peaks will be discarded.

SIGNIFICANT FIGURES *

The term "significant figure" is used, sometimes rather loosely to describe a judgment of the reportable digits in a result. When the judgment is not soundly based, meaningful digits are reported. On the other hand, proper use of significant figures gives an indication of the reliability of the analytical method used.

* From "Handbook for Analytical Quality Control in Water and Wastewater Laboratories"*be done within the limits of the given laboratory operations to improve these values. If more significant figures are needed, a further improvement in method or selection of another method will be required.

Once the number of significant figures obtainable from a type of analysis is established, data resulting from such analyses are reduced according to set rules for rounding off.

The following discussion describes the process of retention of significant figures.

A number is an expression of quantity. A figure or digit is any of the characters 0, 1, 2, 3, 4, 5, 6, 7, 8, 9, which, alone or in combination, serve to express a number. A significant figure is a digit that denotes the amount of the quantity in the particular decimal place in which it stands. Reported analytical values should contain only significant figures. A value is made up of significant figures when it contains all digits known to be true and one last digit in doubt. For example, if a value is reported as 18.8 mg/l, the 18 must be firm while the 0.8 is somewhat uncertain, but presumably better than one of the values 0.7 or 0.9 would be.

The number zero may or may not be a significant figure depending on the situation.

Final zeros after a decimal point are always meant to be significant figures. For example, to the nearest milligram, 9.8 g is reported as 9.800 g.

Zeros before a decimal point with nonzero digits preceding them are significant. With no preceding nonzero digit, a zero before the decimal point is not significant.

If there are no nonzero digits preceding a decimal point, the zeros after the decimal point but preceding other nonzero digits are not significant. These zeros only indicate the position of the decimal point.

Final zeros in a whole number may or may not be significant. In a conductivity measurement of 1,000 umho/cm, there is no implication by convention that the conductivity is 1,000 ± 1 umho. Rather, the zeros only indicate the magnitude of the number.

A good measure of the significance of one or more zeros interspersed in a number is to determine whether the zeros can be dropped by expressing the number in exponential form. If they can, the zeros may not be significant. For example, no zeros can be dropped when expressing a weight of 100.08 g in exponential form; therefore the zeros are significant. However, a weight of 0.0008 g can be expressed in exponential form as 8×10^{-4} g so the zeros are not significant. Significant figures reflect the limits in accuracy of the particular method of analysis. It must be decided whether the number of significant digits obtained for resulting values is sufficient for interpretation purposes. If not, there is little that can

Rounding Off Numbers

Rounding off of numbers is a necessary operation in all analytical areas. It is automatically applied by the limits of measurement of every instrument and all glassware. However, when it is applied in chemical calculations incorrectly or prematurely, it can adversely affect the final results. Rounding off should be applied only as described in the following sections.

Rounding-Off Rules

If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded off to 11.44.

If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded off to 11.45.

If the figure following those to be retained is 5, and if there are no figures other than zeros beyond 5, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded off to 11.44, while 11.425 is rounded off to 11.42.

STANDARD OPERATING PROCEDURES FOR ELECTRONIC BALANCES

Summary

Analytical balances of various capacities and operational mode are calibrated annually by a licensed specialist and officially recorded as verification of performance.

Stickers documenting the calibration are placed on the balance noting the calibration date as well as the date of the next calibration. Certificates of calibration are maintained in the QA/QC department.

Balances are checked with known calibration weights before using. If the values of the calibration are not within limits, the balance will be calibrated per manufacturers instructions. Two examples are provided

The following is a list of check weights used to verify accuracy.

A. Electronic toploaders, (example, Ohaus GT400)

1. Allow a warm up time of 30 to 60 minutes after plugging in the balance.
2. Center bubble level indicator of top loader using the leveling screws.
3. Using kimwipe and/or balance brush, remove any particulates from balance pan.
4. Turn power switch to "1"; the toploader will run through an "auto check".
5. A small green circle "o" appears in the bottom left corner if the system checks out.
6. Press tare once. The display will read 0.00g.
7. Calibration
   a. Calibrate using weights which span the anticipated weighing episode.
   b. Place calibration weight in center of pan using forceps (never touch weights with fingers).
   c. Read and record reading from balance.
   d. Repeat steps "a" and "b" using a second weight.
   e. Document all readings in the daily calibration log.

      NOTE: Reading is stable when "g" appears in the display.
   f. Note control limits recorded in the balance log. Should the weights be outside the control limits, stop the calibration and notify your supervisor or QA Officer.
8. Top loader is now ready to use.
   a. Keep pan as clean as possible.
   b. To tare:

      After reading is stable, press tare and display will read all zeros.

B. Electronic Analytical Balance, OHAUS Model AS120 (reference OHAUS Operating Manual for further detail).

1. If balance has been unplugged, allow 60 minutes for warm-up.
2. Check and center bubble level using leveling screws. 3. Briefly press ON/TARE to tare balance; first 8.8.8.8.8.8.8.8. then 0.0000 will appear.
4. Holding down control bar, allow instrument to display program setting.
   a. Settings for Stringflow are:

mg	160
Int.	2
ASd	2

   Any variation should be noted and balance reprogrammed to the above settings.
5. ***Calibration of Balance:***
   a. Calibrate using weights which span the specified capacity of balance
   b. Remove all objects from pan and close doors.
   c. Hold down ON/TARE until CAL appears.
   d. Release ON/TARE; SPAN will appear
      (1) Press OFF MODE and display will show LIN.
      (2) Press ON/TARE to start the linearity calibration procedure. When ON/TARE is released, C 0 gwill be displayed, indicating that no weight should be in the pan.
      (3) Press ON/TARE. The display will show -C- followed by the value of the weight which must be placed on the pan.
      (4) Place required weight in pan.

(5) Press ON/TARE. The display will show -C- momentarily, then C followed by the next weight to be placed on the pan. Do not disturb balance when -C- is displayed.

(6) Place required weight on the pan, then press ON/TARE. The display will show -C- while the balance recalibrates. When the weight on the pan is displayed along with the current indicator, the balance is recalibrated.

(7) Repeatedly press OFF mode until END is displayed.

(8) Press ON/TARE. When released, the balance will return to normal weighing opterations.

6. *Taring*
   a. Place container on pan.
   b. Press control bar once to tare, all zeros will appear. Balance is now ready for weighing.

Maintenance for the balances is documented with a sticker on the balance dating the service and the next service due date.

STANDARD OPERATING PROCEDURES FOR PH METER (ORION 720)

A. Preliminary Operation

1. General
   a. Allow instrument sufficient time to warm up after turning it on (minimum 15 minutes).
   b. Ensure that all electrode connectors are securely fastened.
   c. Place instrument on "**standby**" when not in use.
   d. Allow samples and buffers to reach uniform temperature before taking measurements.
   e. Stir both buffer and sample solutions •while measurement is being made.
   f. Rinse electrode with DI water between measurements.
   g. Allow time for stabilization of reading, try to maintain same time frame for each sample and buffer.
   h. Calibrate unit.
2. Probe care:
   a. Check and, as needed, add filling solution.
   b. Blot dry only; never wipe.
   c. Store electrode in holding solution when not in use.

B. Calibration:

1. 2-buffer standardization with manual temperature compensation: This step should be done prior to sample analysis, once per day.
   a. Engage "standby" push button (if not on).
   b. With laboratory thermometer, take temperature reading of buffer (pH-7).
   c. Adjust "temperature degree C" control to temperature of buffer solution.
   d. Place electrode in buffer (pH-7).
   e. Engage "pH" push button and place "slope" control in the off position.
   f. Disengage "standby" push button.
   g. Adjust zero controls until digital display reads value of 7.000.
   h. Engage "standby" push button.
   i. Rinse and place electrode in second standard buffer. (This must bracket sample pH value and may not exceed ±3 pH units in range).
   j. Note control limits recorded in the balance log. Should the weights be outside the control limits, stop the calibration and notify the supervisor or QA Officer.
   k. Repeat step "h" using a 2nd weight.
   l. Note % slope reading in notebook (acceptable limit 100% ± 15%).

C. General Information and Documentation

1. pH meter log is to be updated daily when meter is used

2. Maintenance should be dated and noted under "comments" in the pH meter log (see attached example).
3. Aliquots of pH buffers should be replaced at a minimum once per week.
4. pH buffers commercially obtained are dated upon receipt and date of expiration documented.
5. pH buffers commercially obtained must show traceability to NBS standard buffer.

STANDARD OPERATING PROCEDURE FOR CONDUCTIVITY METER (HACH 2711)

A. Preliminary Operation

1. General

a. Adjust meter zero (if necessary) by turning the bakelite screw on meter face so that the meter needle lines up with the zero on the conductivity scale when meter is in "off" position.

b. Turn the "mode" control to redline, adjusting the "redline" control so the meter needle lines up with the redline on the meter face. If you cannot line up the needle with the redline, replace the batteries.

c. Cell check - turn "mode" control to 100x or 10x scale and depress cell button, meter reading should be <2%.

d. Sample depth must be sufficient to cover temperature probe.

e. At time of standard preparation, an aliquot of the Reagent water used should be saved for calibration, (reference step 2.c below)

NOTE: Conductivity will increase with time and exposure to air.

f. Allow standard and samples to reach uniform temperature before taking reading.

g. Always leave meter "off" when not in use to conserve battery.

B. Probe

1. Use

a. Do not touch the electrodes inside probe.

b. Avoid obstructions and when possible, allow 2 inches of clearance from solids.

c. Avoid metallic objects (minimum 6 inches if possible).

d. To ensure flow of sample over electrodes, gently raise and lower the probe several times while taking reading.

2. Cleaning and Maintenance

a. Rinse well with DI water between samples

b. Store probe in DI water when not in use

c. Soak probe in 10 parts isopropyl alcohol/ 10 parts water/1 part concentrated HCl solution for 5 minutes and rinse with DI water once a month, or whenever cell test indicates high reading. If reading is still high, consult reference manual.

3. Calibration

a. Meter: Reference A.1 section a, b, and c

b. Temperature reading: check internal thermometer daily using a second thermometer (i.e., calibrated thermometer) acceptable difference <1°.

(1) Calculation

(I-E) = difference

I = internal thermometer reading in C°

E = 2nd thermometer reading in C°

c. Conductivity reading for accuracy check:

(1) Place probe in Reagent water aliquot stored at time of standard preparation (see A.1.e).

(2) Measure and set temperature using internal thermometer located in probe.

(3) Read conductivity using 1x scale (fresh Reagent water should have conductivity reading of 0, if >20, prepare new standard and Reagent Water aliquot).

(4) Rinse probe and place in 0.745 g KCl/kg Reagent Water standard solution.

(5) Take conductivity reading

* Conductivity reading to be taken on lowest scale

Example: Use X1 scale for less than 500

Use X10 scale for 500 thru 5000

(6) Subtract Reagent water reading from KCl standard reading.

(7) Match temperature and compare actual reading to attached table.

(8) If reading is not within ± 1.5%, notify the Laboratory Manager.

(a) Calculation of percent:

$$\frac{C^{1} - CR \times 100}{CT}$$

CT = Conductivity in umhos/am from table

CR = Conductivity in umhos/am read from meter

C. General Information and Documentation

1. Conductivity log is to be updated daily whenever meter is used.
2. Maintenance should be dated and noted under "comments" in the conductivity log.

STANDARD OPERATING PROCEDURES FOR MISCELLANEOUS EQUIPMENT

A. Refrigerators/Freezers

1. The temperature in all the refrigerators shall be maintained at 4° C (± 2 degrees). In cases where temperatures are out of these limits, the thermostat will be adjusted accordingly with the laboratory manager's approval. If a power failure or some mechanical problem (i.e., compressor) causes the limit to be exceeded, the following actions shall be taken:

   a. <u>If due to power failure ("brown-out")</u>

      Pack all refrigerators with frozen blue ice and/or dry ice* and keep doors closed until the power is restored.

      *Dry ice can be purchased from local supplier.

   b. <u>If due to a mechanical problem</u>

      For repair service, notify a contractor to have it repaired.

      Notify Laboratory Manager. Pack refrigerators with frozen blue ice and/or dry ice, if necessary, until repairs are completed.

2. Temperature must be read daily for all refrigerators and freezers and recorded in the appropriate refrigerator log.

B. Ovens

1. Oven temperatures will be maintained at the required temperature ± 2° C at the operating range of 60-300°C. Above 300°C, temperature will be maintained ± 10°C. If the temperature is found to be out-of-control during analysis, the results of that analysis will not be reported. The analysis will be repeated after the oven has stabilized for 8 hours.

   If stable oven temperature cannot be maintained because of electrical problems, the following should be done:

   a. Notify Laboratory Manager, or if not available;

   b. Call an electrical contractor and arrange for service.

2. Ovens that are set at a specific temperature must have their temperatures read and recorded daily while in use.

C. Desiccator

1. Desiccant must be checked daily and changed when initial color change is first noted (i.e., blue to pink).

D. Fume Hoods

1. Fume hoods flow rates are checked every four months at nine points in each hood for one minute at each point. If the flow rates drop for any reason, the filters for the hoods will be checked and replaced.

E. Steam Baths

1. Steam baths are maintained daily by keeping water level at a certain level and using DI water at all times. Generally they are drained and cleaned weekly.

STANDARD OPERATING PROCEDURE FOR GEL PERMEATION CHROMATOGRAPH (GPC) MAINTENANCE

A. Gel Permeation Chromatograph Autoprep 1002A Maintenance

Routine maintenance is employed by each concerned personnel prior to use (i.e., sample batch run). The following clean-up procedures are followed to help minimize laboratory contamination.

1. Disconnect the GPC column from the system. Seal-off the GPC column to keep Biobeads SX-3 in a moist state by connecting the inflow and outflow tubings together.
2. Solvent Cycle Clean-up:

   The following solvents are pumped to the GPC system (i.e., 23 loops) for a period of 30 minutes per loop to flush away residues built-up in the system. It is pumped through the GPC system in the order of:

   a. Chlorobutane for purging
   b. Acetone (pesticide grade)
   c. Methylene Chloride (Pesticide Grade)
3. If the GPC column appears to be dirty, repack the column.

STANDARD OPERATING PROCEDURE FOR SONICATOR TUNING

Each time a new converter, probe, cup horn, tip, microtip, or accessory is used, the power supply is tuned using the following procedure:

1. Move the switch above the tuning control to the DOWN POSITION.
2. Ensure that the probe or microtip is not immersed in the solution and that it does not come in contact with anything. If a cup horn or flow through cell is used, make sure that it does not contain any water.
3. Set TIMER to HOLD.
4. Set OUTPUT CONTROL to "10" (to "4" when using a microtip or extender).
   CAUTION When using a microtip, never allow the tip to vibrate in air for more than 10 seconds, and do not set the OUTPUT CONTROL above "5". Ignoring these instructions will cause the microtip to fracture.
5. Momentarily hold down ON/OFF/TUNE switch to TUNE and rotate the tuning control clockwise or counterclockwise until a **minimum** (not maximum) reading (usually less than 20) is obtained on the power monitor. If minimum reading (sometimes referred to as null) cannot be obtained, the probe, cuphorn, tip, microtip, or accessory is loose or out of resonance, or the power supply or convertor requiring servicing. A loose probe will usually generate a loud piercing sound.

   NOTE If minimum reading cannot be obtained, check unit without the probe to ascertain whether the power supply or probe is at fault.
6. Set OUTPUT CONTROL to "4".
7. Release ON/OFF/TUNE Switch.
8. With a dual 500 watt Ultrasonic Processor, if two converters are going to be used simultaneously, connect the second converter cable to connector.
9. Document all tuning in the Sonicator Tuning Log.

STANDARD OPERATING PROCEDURE FOR CALIBRATING THERMOMETERS

Thermometers are calibrated quarterly against an NBS thermometer using the following procedure:

1) Install the NBS in the same environment as the thermometer in question (i.e., cold storage refrigerator, oven, etc.)

2) Should the thermometer in the cold storage be in glycerin, verify that the NBS thermometer is in the same solution.

3) Allow the thermometers to equilibrate.

4) Read both thermometers and record in the calibration log.

5) Verify that the thermometer in question is within the ±0.2 acceptance limit.

6) Should the temperature be outside this limit, the thermometer should be replaced with a new calibrated thermometer.

LABORATORY CORRECTIVE ACTION PLAN FOR POTENTIAL ANALYTICAL PROBLEMS

In the following paragraphs,
P = Problem, and
A = Action to be taken

Sample Receipt, Log-in, and Labeling

P: Sample containers received broken and/or seal not intact.

A: Notify Project Officer

P: Sample cannot be located (i.e., misplaced) either intra- or interlaboratory.

A: Notify Project Officer

P: Samples received without proper refrigeration or preservation.

A: Notify Project Officer

P: Illegible sample numbers or label missing from sample containers.

A: Notify Project Officer

P: No instructions received with samples (i.e., list of analytes/analyses to be performed).

A: Notify Project Officer

P: Samples received in nonprotected containers (i.e., not in proper sample containers, samples for VOA analysis not contained in septum top vials.

A. Notify Project Officer

P: Physical characteristics different than those on sampling sheets (i.e., two phases instead of one)

A: Notify Project Officer

P: Shipment container received damaged upon arrival

A: Notify Project Officer

P: Chain-of-Custody document does not match information indicated on sample label and containers received.

A: Notify Project Officer

P: Samples received past the holding time requirement (e.g., nitrates—24 hours).

A: Notify Project Officer

Sample Refrigeration and Preservation

P: Field chain-of-custody sheet indicated that samples were preserved contrary to the protocol or analytical plan.

A: Notify Project Officer

P: No indication on the chain-of-custody or sample containers that the sample was preserved, or how.

A: Notify Project Officer

P: Drastic change in physical characteristics upon preservation in the laboratory.

A: Notify Project Officer and QA Officer

P: Discovery of sample storage (i.e., refrigeration) malfunction

A: Notify Project Officer and QA Officer

P: Discovery that sample has frozen with or without breakage.

A: Notify Project Officer and QA Officer

Analytical Method

P: If at any time you are not in agreement with the method to be used or some portion of the method.

A: Notify Project Officer and QA Officer.

Sample Preparation

P: Loss of sample or unusual behavior during pH adjustment.

A: Notify Project Officer and Supervisor.

P: Knowledge of making incorrect spike.

A: Notify Project Officer and Supervisor.

P: Portion of solvent lost during sample concentration (i.e., KD or N_2 blowdown).

A: Notify Project Officer and Supervisor

P: Sample or extract loss due to glassware breakage

A: Notify Project Officer and Lab Manager

P: A lower than expected percentage of the solvent is recovered after extraction (<85%).

A: Notify Project Officer and Supervisor

P: Unable to reduce the extract volume to desired level or final volume

A: Notify Project Officer and Supervisor

Extract Storage

P: Noticable loss of solvent after storage

A: Notify Project Officer and QA Officer

P: Extract storage is past holding time for analysis (i.e., past 40 days for BNA/Pesticide).

A: Notify Project Officer and QA Officer

P: Noticeable change in physical characteristic.

A: Notify Project Officer, QA Officer, and Lab Manager

P: Label or labels have come off of the storage container.

A: Notify Lab Manager

P: Not enough information is on the label

A: Notify Project Officer and Lab Manager

Standard Preparation

P: Doubt as to purity of the standard material

A: Notify Lab Manager or QA Officer

P: Material does not appear to go completely into solution

A: Notify Project Officer, Lab Manager, or QA Officer

P: Confusion over whether the right compound was added or not

A: Start over from the step you are sure of.

P: Question whether standard (stock or working) is "too old" (expired).

A: Check expiration of the standard, if available. If not, check SOP on standard expiration. ALSO notify Lab Manager or QA Officer

P: Confusion over some dilution from the standard stock solution

A: Start over from the step you are sure of.

Instrumental Analysis

P: Injection of solvent blank produces erratic baseline and/or noise

A: 1) Reinject another solvent blank, if due to bad injection. 2) Check instrument operating conditions. 3) Do corrective maintenance.

P: Injection of multiple components standard produce poor separation or fewer peaks elute than components added.

A: 1) Check column for degradation. 2) Check instrument operating conditions. 3) Do corrective maintenance.

P: Chromatographic peaks ahve severe tailing.

A: 1) Check column for degradation. 2) Check instrument operating conditions. 3) Do corrective maintenance.

P: Multiple standard injections indicate poor instrument and/or analyst precision

A: 1) Check column for degradation. 2) Check instrument operating conditions. 3) Do corrective maintenance.

P: Calibration curve is not linear.

A: 1) Redo calibration with the problematic standard concentration. 2) Redo standard prepration. 3) Recalibrate.

P: Loss of greater than 10% of the sensitivity during the work day is experienced.

A: Check instrument operating conditions.

P: Knowledge that a bad injection has been made.

A: Mark the chromatogram and inject the sample again.

P: Calibration has been performed too infrequently.

A: Notify Lab Manager and QA Officer

P: Retention items begin to change.

A: 1) Check instrument operating conditions. 2) Do column maintenance. 3) Rerun the standard. 4) Recalibrate.

Data Review

P: The recovery of material from spiked water or a QA/QC sample is not within the limits set prior to analysis.

A: Notify the Project Officer.

P: The data is contrary to that expected (historical background does not agree).

A: Notify QA Officer

P: Data review is not done within a day of the analysis, so the corrective changes can be quickly made

A: Notify Lab Manager, Project Officer, or QA Officer

P: Calibration mistake is discovered after data have been reported.

A: Notify Lab Manager and Project Officer or QA Officer.

CHROMATOGRAPHY SECTION — PREVENTIVE MAINTENANCE

MAINTENANCE	FREQUENCY

GASES

- molecular-sieve filters, adsorbent cartridges ..... as needed
- moisture traps/oxygen traps ..... as needed
- gas pressure level (psi) ..... when column maintenance performed
- regulators ..... as needed
- copper line connections, check for gas leaks ..... whenever cylinder changed or work is done on gas lines/traps
- purity of gases used ..... upon receipt of gas order
- gas supplies ..... daily

COLUMNS

- change of glasswool inserts for packed column ...as needed or when column head re-packed
- bake-out temperature frequency .....at end of analysis sequence
- when to change to a new column (i.e., previous column to a new column whether packed or capillary) .....as needed

AUTOSAMPLERS

- flushing of compressed air linesfor the autosampler ..... daily
- alignment of the autoinjector syringe to GC inlet as needed/when syringe is replaced
- autoinjector syringe check for needle/or plunger needle/or plunger ..... as needed
- lubrication of gears .....not required
- replacement of solvent vials used for solvent flush .....as needed
- discard or removal of sample vials that were injected .....daily

AUTOSAMPLER (TEKMAR ALS)

- check gas flow connections .....at installation and when any hardware is .....replaced or changed
- check connection to the GC .....at installation
- check for leaks - glassware mounts of th esampler, fitting, unions sample valve .....at installation and when hardware .....is replaced or changed
- cleaning of the samplers (i.e., fritted disc, sampler, needle spargers, purging device .....every six months or as needed.

RECORDERS/INTEGRATORS

- check supplies (ink, printheads, chart paper) ....weekly
- replacement of printhead/ink .....as needed
- cleaning of pen carriage rods .....as needed

HALL 700A SYSTEM

- check all dials, lights (vent light), and controls for proper function .....daily
- solvent module .....daily
- solvent replacement frequency .....every six months or as needed
- solvent level check .....weekly
- solvent flow rate (solventreplacement, pump shut down) .....when maintenance performed
- motor pump for proper function .....daily
- ion exchange resin .....monthly
- frequency of packing or replacement .....yearly
- replacement of nickel reaction tube .....as needed

PHOTOIONIZATION DETECTOR (PID MODEL 703)

- cleaning of lamp window ..... as needed
- cleaning of detector .....as needed
- replacement of detector lamp .....as needed

GAS CHROMATOGRAPHS

1. Check for gas leaks

- gas lines/column connections .....at installation, during routine maintenance
- column joint with injection port .. ..at installation, during routine maintenance
- GC connection to gas lines .....at installation and when gas lines are changed .....to the instrument
- septum (auto-injection port) replacement .....as needed

2. Injection port

- cleaning .....as needed
- replacement of glass liner insert .....as needed
- replacement of charcoal tube filter for exhaust/vent monthly
- replacement of brass seal (swagelok/ferrules) for column connection .....as needed
- gas flow checks .....at time of routine column\injection .....port maintenance
- temperature checks .....daily

3. Detectors

- cleaning/rebuilt .....as needed
- radioactive leak check (wipe test) .....every six months
- column pressure (psi) .....at time of routine column\injection .....port maintenance
- check panels, light, dials, controls for proper function .....daily

GENERAL

- cleaning and maintenance of bench tops .....monthly
- check supplies ..... weekly
- logbook entries for maintenance and temperature programs, injection logs, etc. .....as required

Solvent Disposal

Solvents from sample extraction or sample prep shall be disposed of by incineration through the use of a properly licensed/permitter facility. All drums shall be manifested for transportation; it is essential to have the waste segregated by type.

FIGURE D.2
Hazardous Waste Drum Sticker

A. Chlorinated solvents i.e., Freon/Methylene Chloride

B. Hydrocarbons - Toluene/Hexane/Cyclohexane Segregation shall begin within laboratory, small two gallon containers shall be placed within each laboratory area. These shall be of proper type to receive solvent and properly labeled as to type and with any hazard codes.

At the end of each work day these containers shall be emptied into a fifty gallon drum located in a secure area behind the walk-in. All waste solvent cans shall have a minimum of *two gallon* capacity and have a self closing lid to reduce the amount of volatile loss. The only time the lids are to be opened are when the can is in use. Each container shall be equipped with a integral flame arrestor and a pressure relief mechanism. One suggested supplier is Lab Safety *catalog number 0891.*

A log shall be maintained (see Figure D.1) on each drum as to the date it was put into service and when it is full. This log shall become part of the manifest and a copy maintained with the lab for record of disposal. Each drum shall be of the proper type required and labeled with type of waste/hazard codes and our name and address, date that was in service and a unique sequential identification (see Figure D.2). These drums shall be used only for solvent disposal. Records of solvent disposed shall be the responsibility of the extraction lab supervisor. Any deviation from procedure or problems associated with disposal of solvent shall be documented with a corrective action report. All waste drums shall meet DOT requirements and be lined with plastic or coated with phenolic resin. The use of this type of drum is to reduce potential leaking from corrosion if water or corrosive material are inadvertently added to the drums. Drums shall be ground at all times when being filled as well as the waste solvent can to prevent static sparks(see figure D.3). After each use drums shall be closed using a non-sparking wrench (see figure D.4)(Lab Safety Supply Catalog number C4762). After tightening bungs, place cover (Lab Safety supply catalog number C 2367) over drum to prevent rain water from accumulating in top of drum (see figure D.5). It is mandatory this secure area be kept organized and locked.

The waste stream needs to be periodically characterized by analysis and accurate record maintenance. Based upon our knowledge of the waste stream and the analysis all manifest shall be completely filled out.

The secured area shall be deemed such and sign posted (Labeled Hazardous Waste, No Smoking and Emergency Phone Number—See figure D.6). Any noncompatible waste should be stored as far away from each other as possible.

All drums should be moved using an approved drum dolly (see figure D.7) (Lab Safety supply catalog number C4087) protective eyewear and gloves should be worn when filling or transporting any of the waste.

Emergency Spill Kit containing vermiculite should be available as an absorbent in case of spill along with Spill Dikes (Lab Safety Supply catalog number C7412-2) to contain any spill in a localized area (see figure D.8).

An emergency response kit shall be maintained near or in the containment area. This kit shall contain protective gloves, respirator with assorted cartridges (i.e., solvent vapor and gas) protective clothing and goggles.

HAZARDOUS WASTE STORAGE & INSPECTION PROCEDURE

Scope:

Southwest Laboratory of Oklahoma, Inc. utilizes this procedure to assure proper storage identification and correction of hazardous waste hazards and compliance with all hazardous waste SOP's. It is the responsibility of Management to assure that this procedure is followed.

Procedure:

1. Waste are stored in a designated containment area until periodic removal by waste hauler.
2. Inspections should be completed weekly.
3. The hazardous waste inspection form will be used as a check-list and for documentation of the inspection (see Figure D.9).
4. The inspection form is divided into four sections:
   - General; signs, housekeeping, SOP's followed.
   - Containers; odors present, caps on tight, leaking.
   - Labeling; DOT labels present, hazardous waste label present, flammable label present, drums marked properly, accumulation start date.
   - Safety; necessary safety equipment accessible.
5. The inspection form will be used as a tool for corrective action and follow-up.
6. The inspection form will be filed by the Hazardous Waste Manager.

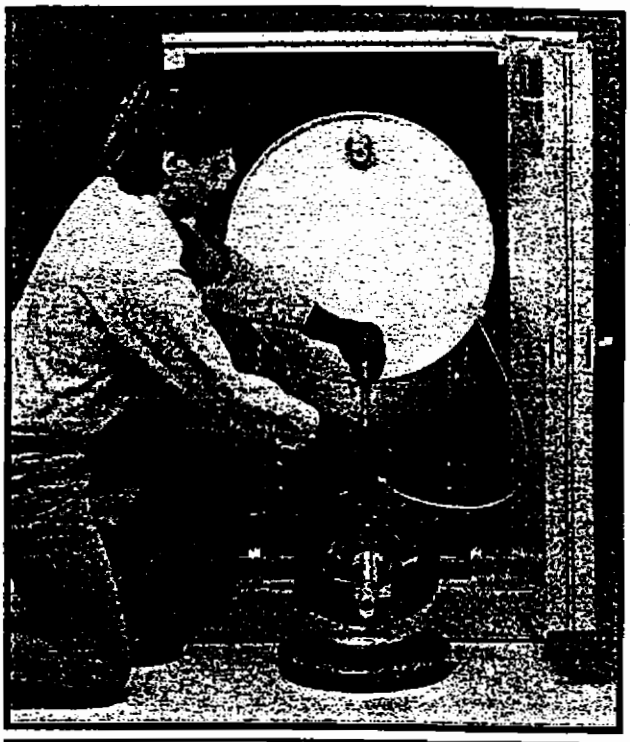


FIGURE D.3
Drum Storage Cabinet

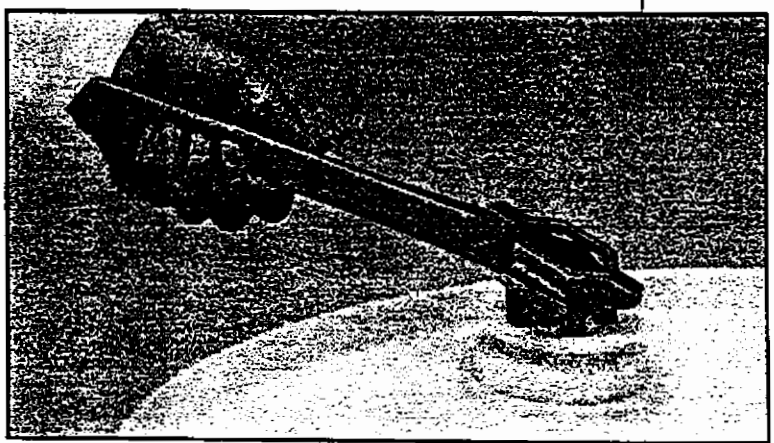
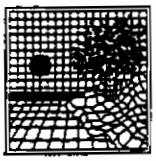


FIGURE D.4
Non-Sparking Drum Plug Wrench

HAZARDOUS WASTE MANIFEST

Scope:

Southwest Laboratory of Oklahoma, Inc. utilizes this procedure to assure that all hazardous waste shipments are manifested properly and in accordance with DOT and Hazardous Waste regulations. It is the responsibility of Management to assure that this procedure is followed.



HAZARDOUS WASTE DISPOSAL RECORD

CAN #:_____ DATE TRANSFERRED:_____

CHLORINATED: ❑ NON-CHLORINATED: ❑ BARREL #:_____

Date	Amount (mls)	Description, Amt of each Constituent	Initials

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 W. ALBANY • BROKEN ARROW, OKLAHOMA 74012 • OFFICE (918) 251-2858 • FAX (918) 251-2599

FIGURE D.1 Hazardous Waste Disposal Record

Procedure:

1. A Hazardous Waste manifest must be completed according to the instructions on the manifest before the wastes can be picked-up for transportation to the disposal site (see Figure D.10).
2. The truck driver must sign and leave two copies of the manifest with us.
3. One copy of the manifest is retained in the Hazardous Waste Manger's file and the other is mailed immediately to the state receiving the waste.
4. Manifest records are maintained for a minimum of three years.

HAZARDOUS WASTE REPORTING REQUIREMENTS

Scope:

Southwest Laboratory of Oklahoma, Inc. utilizes this procedure to assure that all hazardous waste reports are generated according to applicable regulations. It is the responsibility of management to assure that this procedure is followed.

Procedure:

1. All completed manifest shall be filed quarterly with the State. No report is required to be filed with the USEPA.
2. If the completed manifest is not received from the disposal site within 30 days from the date accepted by the initial transporter, and "Exception Report" must be filed with the State within 45 days from the date of shipment. This report must contain the action taken to determine the status of the shipment.

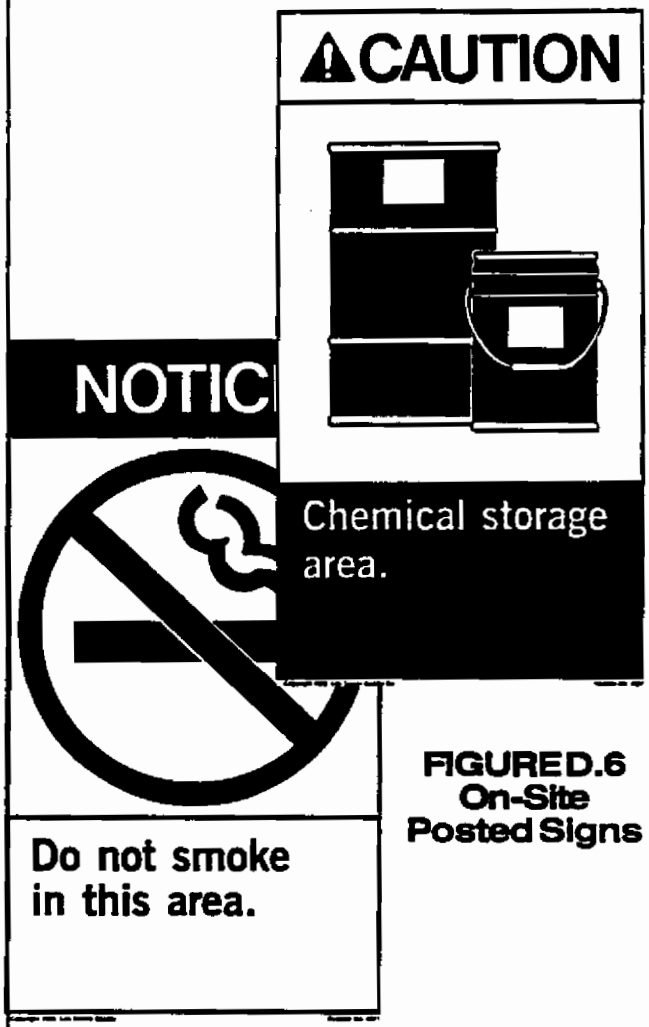


FIGURE D.6
On-Site Posted Signs

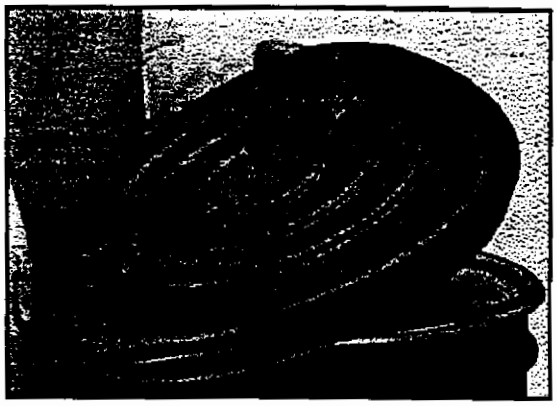


FIGURE D.5
Drum Cover

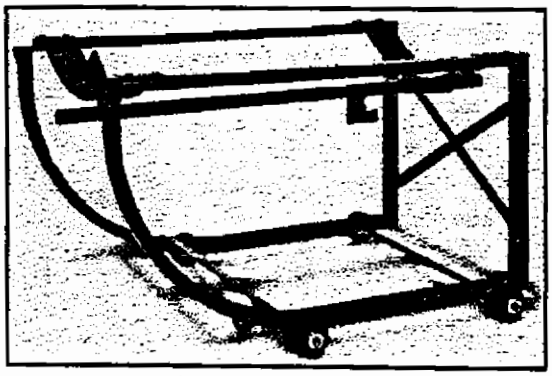


FIGURE D.7
Approved Drum Dolly

FIGURE D.8
Spill Dike

HAZARDOUS WASTE INSPECTION FORM

DATE: _____

TIME: _____

AREA INSPECTED

SATELLITE ☐ BULK STORAGE ☐

AREA INSPECTED	OBSERVATIONS/REQUIRED ACTIONS	DATE CORRECTED
GENERAL		
Housekeeping		
SOP's		
Signs		
CONTAINERS		
Odors		
Caps Sealed		
Leaking?		
LABELS		
DOT/Hzd Wst		
Markings		
Accum Date		
SAFETY		
Signs		
Equipment		
COMMENTS		

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 W. ALBANY • BROKEN ARROW, OKLAHOMA 74012 • OFFICE (918) 251-2858 • FAX (918) 251-2599

FIGURE D.9 Hazardous Waste Inspection Form

STATE OF ARKANSAS
Department of Pollution Control and Ecology
P. O. Box 8913 Little Rock, Arkansas 72219-8913
Telephone 501-562-7444

1

Please print or type. *(Form designed for use on elite (12-pitch) typewriter.)*

Form Approved. OMB No. 2050-0039. Expires 9-30-91

UNIFORM HAZARDOUS WASTE MANIFEST

1. Generator's US EPA ID No. | Manifest Document No.

2. Page 1 of | Information in the shaded areas is not required by Federal law.

3. Generator's Name and Mailing Address

A. State Manifest Document Number: AR- 494389

B. State Generator's ID

4. Generator's Phone ( )

5. Transporter 1 Company Name | 6. US EPA ID Number

C. State Transporter's ID PC ____ H ____

D. Transporter's Phone

7. Transporter 2 Company Name | 8. US EPA ID Number

E. State Transporter's ID PC ____ H ____

F. Transporter's Phone

9. Designated Facility Name and Site Address | 10. US EPA ID Number

G. State Facility's ID

H. Facility's Phone

11. US DOT Description (including Proper Shipping Name, Hazard Class, and ID Number)	12. Containers No.	Type	13. Total Quantity	14. Unit Wt./Vol.	I. Waste No.
a.					
b.					
c.					
d.					

J. Additional Descriptions for Materials Listed Above

K. Handling Codes for Wastes Listed Above

EMERGENCY RESPONSE INFORMATION

If no alternate TSDF, return to generator

15. Special Handling Instructions and Additional Information

16. **GENERATOR'S CERTIFICATION:** I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked, and labeled, and are in all respects in proper condition for transport by highway according to applicable international and national government regulations and Arkansas state regulations.
If I am a large quantity generator, I certify that I have a program in place to reduce the volume and toxicity of waste generated to the degree I have determined to be economically practicable and that I have selected the practicable method of treatment, storage, or disposal currently available to me which minimizes the present and future threat to human health and the environment; OR, if I am a small quantity generator, I have made a good faith effort to minimize my waste generation and select the best waste management method that is available to me and that I can afford.

Printed/Typed Name | Signature | Month Day Year

17. Transporter 1 Acknowledgement of Receipt of Materials

Printed/Typed Name | Signature | Month Day Year

18. Transporter 2 Acknowledgement of Receipt of Materials

Printed/Typed Name | Signature | Month Day Year

19. Discrepancy Indication Space

20. Facility Owner or Operator: Certification of receipt of hazardous materials covered by this manifest except as noted in Item 19.

Printed/Typed Name | Signature | Month Day Year

EPA Form 8700-22 (Rev. 9-88) Previous edition is obsolete.

NOTICE: THE ORIGINAL AND NOT LESS THAN TWO (2) COPIES MUST MOVE WITH THE HAZARDOUS WASTE SHIPMENT. ONCE DELIVERED, THE TREATMENT/STORAGE/DISPOSAL FACILITY MUST RETURN THIS ORIGINAL COPY TO THE GENERATOR.

920-021 (11-90) ST-90-0011 Consolidated Business Forms, Inc. - Bentonville, AR 72712 501-273-3486 24004

FIGURE D.10 Uniform Hazardous Waste Manifest

Appendix E
Laboratory Control Limits

PARAMETER	METHOD REFERENCE	SW846 METHOD LIMITS	CLP METHOD LIMITS	LABORATORY LIMITS % REC	LABORATORY (MAX RPD) WATER/SOIL
VOLATILE ORGANICS	624/8240				
• Surrogate Spikes	EPA/SW846			•	
Toluene d-8 (water)		88-110	88-110	88-110	
Toluene d-8 (soil)		81-117	84-138	81-117	
Bromofluorobenzene (water)		86-115	86-115	86-115	
Bromofluorobenzene (soil)		74-121	59-113	74-121	
1,2-Dichloroethane-d4 (water)		76-114	76-114	76-114	
1,2-Dichloroethane-d4 (soil)		70-121	70-121	70-121	
• Laboratory Control Spikes		water/soil		water/soil	water/soil
Acetone		53-146/30-160		53-146/30-160	50/59
Bromodichloromethane		48-164/71-124		48-164/71-124	20/14
Benzene		70-134/79-128		70-134/79-128	46/13
Bromoform		34-151/58-119		34-151/58-119	52/37
Bromomethane		50-191/46-161		50-191/46-161	23/36
Chlorobenzene		72-135/80-125		72-135/80-125	14/10
Carbon Disulfide		50-162/52-129		50-162/52-129	31/29
cis-1,2-Dichloroethene		54-153/80-128		54-153/80-128	24/20
cis-1,3-Dichloropropene		34-199/63-123		34-199/63-123	33/29
Chloroethane		32-212/-44-154		32-212/-44-154	28/54
Chloroform		57-152/79-129		57-152/79-129	9/177
Chloromethane		32-161/21-170		32-161/21-170	28/18
Carbon Tetrachloride		37-182/65-140		37-182/65-140	49/21
2-Chloroethylvinyl-ether		73-156/D-146		73-156/D-146	61/45
Dibromochloromethane		43-157/68-120		43-157/68-120	21/25
1,3-Dichlorobenzene		52-164/64-121		52-164/64-121	37/24
1,1-Dichloroethene		54-161/60-141		54-161/60-141	32/16
1,2-Dichloropropane		40-182/81-122		40-182/81-122	32/17
Ethylbenzene		55-147/82-123		55-147/82-123	17/19
2-Hexanone		27-176/20-181		27-176/20-181	97/60
Methylene Chloride		52-168/77-141		52-168/77-141	43/21
4-Methyl-2-Pentanone		23-208/33-163		23-208/33-163	62/54
m,p-Xylene		55-151/81-125		55-151/81-125	16/21
o-Xylene		68-144/72-126		68-144/72-126	29/14
Styrene		64-147/71-129		64-147/71-129	27/13
Trichloroethene		67-149/71-134		67-149/71-134	38/21
Trichlorofluoromethane		39-181/70-132		39-181/70-132	28/25
trans-1,2-Dichloroethene		74-162/72-133		74-162/72-133	35/21
trans-1,3-Dichloropropene		10-164/48-141		10-164/48-141	42/41
Toluene		75-140/80-122		75-140/80-122	15/11
Tetrachloroethene		61-141/80-125		61-141/80-125	24/21
Vinyl Acetate		22-122/17-153		22-122/17-153	60/72
Vinyl Chloride		65-167/31-168		65-167/31-168	25/26
1,1-Dichloroethane		55-159/75-129		55-159/75-129	22/19
1,1,1-Trichloroethane		38-179/66-140		38-179/66-140	51/45
1,1,2,2-Tetrachloroethane		14-157/49-127		14-157/49-127	95/78
2-Butanone		42-173/16-130		42-173/16-130	29/91
1,2-Dichlorobenzene		62-135/53-120		62-135/53-120	39/16
1,2-Dichloroethane		59-151/81-132		59-151/81-132	26/19
1,1,2-Trichloroethane		40-179/68-120		40-179/68-120	31/28
1,4-Dichlorobenzene		51-172/63-112		51-172/63-112	46/32

*CLP Limits used due to excessive in-house generated limits.

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PARAMETER	METHOD REFERENCE	SW846 METHOD LIMITS	CLP METHOD LIMITS	LABORATORY LIMITS % REC	LABORATORY (MAX RPD) WATER/SOIL
• Matrix Spike/Duplicate (RPD)*				water*/soil	*
1,1-Dichloroethane		59-155	61-145/59-172	61-145/77-135	14/22
Trichloroethene		71-157	71-120/61-137	71-120/79-100	14/24
Benzene		37-151	76-127/66-142	76-127/77-135	11/21
Toluene		47-150	76-125/59-139	76-125/77-134	13/21
Chlorobenzene		37-160	75-130/60-133	75-130/92-115	13/21
BTEX	8020				
• Surrogate Spike	EPA SW-846				
Bromofluorobenzene		65-135		65-135	
• Matrix Spike/Duplicate (RPD)					
Benzene		39-150		39-150	20
Toluene		46-148		46-148	20
Ethyl benzene		32-160		32-160	20
Xylenes		77-145		77-145	20
BNA	625/8270				
• Surrogate Spikes	EPA/SW846			water/soil	
Nitrobenzene-d5		35-114/23-120	35-114/23-120	47-102/40-98	
2-fluorobiphenyl		43-116/30-115	46-116/30-115	46-86/34-125	
p-terphenyl-d14		33-141/18-137	33-141/18-137	66-100/18-137*	
Phenol-d5		10-94/24-113	10-110/24-113	46-86/33-96	
2-fluorophenol		21-100/25-121	21-110/25-121	21-100*/32-82	
2,4,6-Tribromophenol		10-123/19-122	10-123/19-122	43-115/19-122*	
2-Chlorophenol-d4			33-110/20-130		
1,2-Dichlorobenzene-d4			16-110/20-130		
• Laboratory Control Spikes		water/soil		water/soil	water/soil
Phenol		28-104/35-115		28-104/35-115	40/37
2-Chlorophenol		21-106/34-115		21-106/34-115	29/38
1,4-Dichlorobenzene		14-110/33-201		14-110/33-102	29/38
n-Nitroso-di-n-propylamine		19-128/36-124		19-128/36-124	29/40
1,2,4-Trichlorobenzene		17-109/43-106		17-109/43-106	27/37
4-Chloro-3-mehtylphenol		23-115/30-124		23-115/30-124	33/33
Acenaphthene		26-137/41-113		26-137/41-113	27/37
4-Nitrophenol		20-135/25-147		20-135/25-147	33/38
2,4-Dinitrotoluene		28-129/38-122		28-128/38-122	28/28
Pentachlorophenol		25-134/28-133		25-134/28-133	46/39
Pyrene		28-139/26-126		28-139/26-126	16/46
• Matrix Spike/Duplicate (RPD)*				water*/soil	*
1,2,4-Trichlorobenzene		44-142	39-98/38-107	39-98/58-93	28/23
Acenaphthene		47-145	46-118/31-137	46-118/59-89	31/19
2,4-Dinitrotoluene		39-139	24-96/28-89	24-96/56-97	38/47
Pyrene		52-115	26-127/35-142	26-127/44-109	31/36
N-Nitroso-Di-n-Propylamine		13.6-197.9	41-116/41-126	41-116/45-116	38/38
1,4-Dichlorobenzene		20-124	36-97/28-104	36-97/23-75	28/27
Pentachlorophenol		14-176	9-103/17-109	9-103/31-167	50/47
Phenol		5-112	12-110/26-90	12-110/50-71	42/35
2-Chlorophenol		23-134	27-123/25-102	27-123/39-83	40/50
4-Chloro-3-Methylphenol		22-147	23-97/26-103	23-97/60-87	42/33
4-Nitrophenol		0-132	10-80/11-114	10-80/73-102	50/50

*CLP Limits used due to excessive in-house generated limits.

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PARAMETER	METHOD REFERENCE	SW846 METHOD LIMITS	CLP METHOD LIMITS	LABORATORY LIMITS % REC	LABORATORY (MAX RPD) WATER/SOIL
PESTICIDE/PCB	608/8080				
• **Surrogate Spike**	EPA/SW846				
Dibutyichlorendate (water)			60-150/60-150	—	
Dibutyichlorendate (soil)			60-150/60-150	—	
Tetrachloro-m-xylene			60-150/60-150	—	
Decachlorobiphenyl			60-150/60-150	—	
• **Laboratory Control Spikes**		**water/soil**		**water/soil**	
Aldrin		38-123/22-119		38-123/22-119	
Dieldrin		40-123/20-123		40-123/20-123	
Endrin		42-145/25-138		42-145/25-138	
g-BHC		34-143/21-153		34-143/21-153	
Heptachlor		39-128/21-145		39-128/21-145	
4,4'-DDT		36-126/45-131		36-126/45-131	
a-BHC		31-142/20-160		31-142/20-160	
b-BHC		43-133/22-147		43-133/22-147	
d-BHC		24-125/23-104		24-125/23-104	
Endrin Aldehyde		47-178/27-160		47-178/27-160	
Endrin Ketone		49-161/23-147		49-161/23-147	
Endosulfan I		46-134/24-121		46-134/24-121	
Endosulfan II		41-149/24-142		41-149/24-142	
Endosulfan Sulfate		36-150/22-126		36-150/22-126	
g-Chlordane		40-137/21-118		40-137/21-118	
Heptachlor Epoxide		39-124/20-119		39-124/20-119	
Methoxychlor		34-141/26-140		34-141/26-140	
4,4'-DDD		70-133/57-160		70-133/57-16	
4,4'-DDE		70-122/50-117		70-122/50-117	
• **Matrix Spike/Duplicate**			**water/soil**	*	*
Gamma-BHC			56-123/46-127	56-123/46-127	15/50
Heptachlor			40-131/35-130	40-131/35-130	20/31
Aldrin			40-120/34-132	40-120/34-132	22/43
Dieldrin			52-126/31-134	52-126/31-134	18/38
Endrin			56-121/42-139	56-121/42-139	21/45
4,4-DDT			38-127/23-134	38-127/23-134	27/50
TRACE METALS (ICP)*	200.7/6010 EPA/SW846				
Aluminum		75-125	75-125	75-125	20
Barium		75-125	75-125	75-125	20
Boron		75-125	75-125	75-125	20
Calcium		75-125	75-125	75-125	20
Cobalt		75-125	75-125	75-125	20
Iron		75-125	75-125	75-125	20
Magnesium		75-125	75-125	75-125	20
Manganese		75-125	75-125	75-125	20
Molybdenum		75-125	75-125	75-125	20
Potassium		75-125	75-125	75-125	20
Silicon		75-125	75-125	75-125	20
Sodium		75-125	75-125	75-125	20
Vanadium		75-125	75-125	75-125	20

*CLP Limits used due to excessive in-house generated limits.

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PARAMETER	METHOD REFERENCE	SW846 METHOD LIMITS	CLP METHOD LIMITS	LABORATORY LIMITS % REC	LABORATORY (MAX RPD) WATER/SOIL
TRACE METALS (FURANCE AA)*	7000 Series SW846				
Arsenic		75-125	75-125	75-125	20
Lead		75-125	75-125	75-125	20
Selenium		75-125	75-125	75-125	20
Thallium		75-125	75-125	75-125	20
Mercury (CV)*	7470 SW846	75-125	75-125	75-125	20
WET CHEMISTRY					
Carbonate	E310.0			80-120	20
Bicarbonate	E310.0			80-120	20
Hydroxide	E310.0			80-120	20
TDS	E160.1				20
Chloride	E300.0			80-120	20
Fluoride	E300.0			80-120	20
Sulfate	E300.0			80-120	20
Nitrate	E300.0			80-120	20
Ortho-Phosphate	E300.0			80-120	20
Nitrate	E352.1			80-120	20
Nitrite	E354.1			80-120	20

*CLP Limits used due to excessive in-house generated limits.

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NAVY QUALITY CONTROL LEVELS

As detailed in the Naval Energy & Environmental Support Activity (NEESA) Document 20.2—047B here are different levels of Quality Control that meet the requirements of the Data Quality Objectives (DQOs) for varios remedial actions. The following table details these levels and the Quality control needed to support them.

TABLE E.1 OVERALL PLAN FOR QC BASED ON TYPE OF SITE

DQO Levels[1]	Type of Site	QC Requirements						
3	Major Non-NPL Level C	PE sample	Laboratory[2] audit	QA Plan review	Use EPA-approved method[3]	Monthly review	10% field duplicates	Review of final data
4	NPL Level D	PE sample	Laboratory[2] audit	QA Plan review	Use CLP procedures	Monthly review	10% field duplicates	CLP validation
5	Non-NPL Level E	PE sample	Laboratory[2] audit	QA Plan review	Use EPA-approved methods.[3] Non-EPA methods for tissue and explosives.	Monthly review	5% Field duplicates	Review of final data

[1]QC criteria for DQO Levels 1 and 2 has not been defined.
[2]All laboratory audits will be performed by the NCR.
[3]Includes methods from SW 846, Amercian Society for Testing Materials, and Federal Register.

CLP = Contract laboratory protocol
PE = Performance evaluation samples
DQO = Data quality objective

R1-8/88

TRIANGLE LABORATORIES of RTP, INC.

QUALITY ASSURANCE MANUAL

UNCONTROLLED COPY

TABLE OF CONTENTS

Sections

Appendices

Section 1

INTRODUCTION

This manual is a description of the quality assurance program employed at Triangle Laboratories of RTP, Inc., referred to hereafter as Triangle Labs or TLRTPI. It is intended to provide employees, accrediting agencies, and clients with the information needed to understand how an effective and economical quality assurance system is maintained at Triangle Labs. The QA Manual is divided into fifteen sections and several appendices. The first three sections pertain to the manual itself. Sections 4 - 7 provide general descriptions of Triangle Labs, including its objectives, policies, facilities, organization, personnel, and services. The remaining sections describe specific quality assurance activities as practiced within different functions or work units. The order of sections 8 - 12 closely follows that of the production process at Triangle Labs. The appendices provide supplemental materials that support the descriptions in the QA Manual sections.

Written procedures for implementing the activities described in this manual may be found within the Standard Operating Procedures (SOP's), which are available for application in widely distributed SOP Manuals. The provisions of this manual are binding upon those laboratory personnel assigned responsibilities described herein. All laboratory personnel must adhere implicitly to the Standard Operating Procedures.

Section 2

AUTHORIZATION

The quality assurance system described in this Quality Assurance Manual has the absolute support of the management at Triangle Labs.

The provision of quality analytical services to our customers has given us an enviable reputation and has made us a leader in the industry. Assuring that we maintain this status in providing quality products to our customers is the responsibility of every member of the laboratory staff. It is hoped that everyone concerned will use this manual as a guide to quality improvement and to maintenance of our current standing as a quality-oriented laboratory.

Signature:	Date:
J. Ronald Hass President and Chief Executive Officer	12/8/94
Patty Ragsdale Quality Assurance Manager	12/6/94
Sarah VanBenthuysen General Manager	12/8/94
Edwin Marti Technical Director, Environmental Division	12/8/94

Section 3

MANAGEMENT OF THE QUALITY ASSURANCE MANUAL

The quality assurance department (Quality Assurance Unit - QAU) bears primary responsibility for the publication and distribution of the Quality Assurance Manual. The manual is submitted for senior management review on a minimum annual basis, the results of which are documented, utilized for revisions, and maintained in QAU files. As major changes are implemented in the quality assurance system, with senior management approval, the Quality Assurance Manual is also revised. The assistance of laboratory management is essential for the publication of the QA Manual, with contributions by departmental supervisors of specific information for incorporation in the manual.

Authorization signatures found in Section 2 of the manual signify senior management review and approval of the Quality Assurance Manual. Organizational changes that affect the positions on this page require that it be revised as soon as practicable. The revision date for this section must be the most recent of any section in the manual, indicating that all revisions have undergone management review.

Document control procedures are applied in the distribution of copies of the Quality Assurance Manual. Controlled copies are serially numbered, with the maintenance of a distribution list. Revisions are distributed to recipients of the manual when necessary and controlled copies are retrieved when personnel changes require that some employees no longer possess the manual. Uncontrolled copies may be issued to persons or organizations outside of Triangle Labs. They are to be so marked ("uncontrolled"), and will not be subject to updates upon revision of the manual.

Upon revision, all text added or changed since the last issue of each section will be marked with a vertical bar in the margin.

Section 4

THE QUALITY ASSURANCE OBJECTIVE, POLICIES AND PROGRAM

The Quality Assurance Objective

The Quality Assurance Objective at Triangle Laboratories of RTP, Inc. is to demonstrate or prove to the users of our products, regulatory agencies, accrediting agencies, senior management, and laboratory employees themselves, that all requirements for the products have been satisfied.

The Quality Assurance Policies

The management of Triangle Labs supports the following policies in order to achieve the Quality Assurance Objective and promote the Quality Assurance Program:

- Standard procedures shall be implemented in order to determine client requirements and to clearly communicate these requirements within the laboratory.
- Organizational emphasis on quality improvement will take place through strong management commitment and leadership, employee empowerment and teamwork.
- A comprehensive quality control system shall be established and maintained in order to verify and assure continued precision and accuracy of analytical results.
- Adequate training on laboratory operations shall be available to all employees whose decisions may affect the quality of laboratory products.
- A comprehensive program of documentation shall be implemented to prove that adequate quality control procedures have been implemented, that accountability has been maintained, and that traceability has been facilitated.
- Measures shall be implemented to ensure that sample integrity is protected.
- Validation studies shall be performed for each analytical method, including extensive evaluations whenever major modifications have been implemented.
- The instrumentation, equipment, and materials used in the production process shall be controlled (i.e., purchased, verified, calibrated, maintained, monitored, and evaluated) to ensure that required standards are met.
- A comprehensive program for data reduction, validation, reporting, and archival shall be implemented.

- Preventive and corrective actions shall be taken to eliminate the causes of potential or actual nonconformance. Emphasis shall be placed on preventive measures in order to reduce the cost of detection and correction of problems.

- Measures shall be implemented in order to meet the requirements set forth by agencies from whom certifications and accreditations have been granted.

The Quality Assurance Program

A comprehensive Quality Assurance Program has been established at Triangle Labs. The program is summarized in an instructional publication which is available to all employees. This document serves to clarify the diverse roles and activities of personnel in the application of quality assurance concepts.

Section 5

LABORATORY DESCRIPTION

Triangle Laboratories of RTP, Inc. and Affiliated Laboratories

The location, mailing address, and phone numbers for Triangle Laboratories of RTP, Inc. are:

Triangle Laboratories of RTP, Inc.
801 Capitola Drive
Durham, North Carolina 27713

P.O. Box 13485
Research Triangle Park, North Carolina 27709

(919) 544-5729
(919) 544-5491 (Facsimile)

Triangle Laboratories of RTP, Inc. is a C Corporation and is not publicly traded. The company has experienced rapid growth since its incorporation in November 21, 1984, resulting in affiliations with several companies. Triangle Laboratories of RTP, Inc. stands alone as a company and is solely responsible for its quality. Affiliated companies include the following:

Triangle Laboratories of Houston, Inc. was incorporated May 4, 1989.

Triangle Laboratories of Columbus, Inc. was incorporated September 25, 1990.

Triangle Laboratories of Atlanta, Inc. was incorporated December 17, 1990.

TLINC is a holding company that was incorporated in January 1, 1992 to provide unifying structure and a means of financial support to the Triangle Laboratories Family.

Facilities and Instrumentation

Triangle Laboratories of RTP, Inc. currently occupies facilities of more than 40,000 square feet. Facilities are divided according to work function, including separate areas for sample receipt; sample, standard, and glassware preparation; sample and data storage; instrumentation; report preparation; quality assurance; shipping; maintenance; and business/management offices.

Analytical instrumentation at Triangle Labs includes one or more of the following: high resolution gas chromatograph/high resolution mass spectrometers (HRGC/HRMS); high resolution gas chromatograph/low resolution mass spectrometers (HRGC/LRMS); gas chromatographs (GC) with electron capture (ECD), flame ionization (FID), and other detectors; AOX/TOX adsorption

module and microcoulometric-titration systems; ion chromatographs (IC); inductively coupled plasma-atomic emission spectrophotometers (ICP) and atomic absorption spectrophotometers (AA).

Well maintained equipment is essential in assuring the timely delivery of complete, high quality analytical data to clients. This is facilitated through a program of regular maintenance for all equipment, equipment redundancy, an ample stock of spare parts, and an inventory of specialized test equipment to support rapid repair when unscheduled maintenance is required. Perhaps the most important feature of our equipment maintenance and repair plan is the availability on our staff of personnel capable of providing instrument service without reliance on off-site service technicians. Procedures and schedules for preventive maintenance are available in several SOP's. Every instrument has a logbook in which both scheduled preventive maintenance and corrective actions are recorded.

Environmental and Security Systems

Triangle Labs provides a secure environment for our employees, guests, clients, samples and analytical data.

Access Our standard procedures require that all doors remain locked except the main entrances during regular business hours. Once access to the lobbies, break rooms, meeting rooms and other low security areas is gained there are secondary combination lock mechanisms. The combination numbers on these locks are changed periodically. Access to the analytical laboratories is normally restricted to Triangle Labs employees. Exceptions will be made in the case of tours of the laboratories by prospective customers and customers who have a need to be present when their samples are analyzed. Visitors are to sign the Visitor Log and are to be accompanied by a Triangle Labs employee at all times. Several rules are applied regarding punch lock entrances. New punch lock combinations are obtained from an employee's supervisor or the receptionist. Combinations are to be given only to active Triangle Labs employees. When accompanied by visitors, employees obscure the punch lock combination from view.

Security Triangle Labs contracts with a national security company to place a guard on site during our off peak hours. In addition, the security alarm at Triangle Labs offers continuous monitoring for smoke, extreme fire related heat, cold room temperatures, motion, and door contacts; battery backup; an automatic dialing feature that calls the alarm company and appropriate authorities when activated, and a panic button that sets off the audible alarm and calls the central station.

Archives Limited access archive facilities are maintained that house all Triangle Labs copies of analytical reports, raw data, inactive logbooks, magnetic tapes and other data which facilitate traceability of analytical results. Steps are taken to ensure continued integrity of the materials kept here.

Chemical Storage and Disposal

All chemicals are stored in appropriate cabinets and are properly disposed of when necessary. All flammable solvents are kept in OSHA and NFPA approved cabinets. Acids are stored in OSHA approved acid cabinets. An authorized waste carrier is contracted to pick up lab waste monthly and dispose of it, usually by incineration, meeting all regulatory requirements. Post-analysis disposition of samples is dependent upon client requests. Remaining sample material may be returned to the client, safely discarded, or archived for a specific period of time.

Environmental Control

The working and storage environments are maintained in a safe and appropriate manner. Heating, ventilation and air-conditioning systems satisfy the needs of personnel, equipment and supplies. Lighting, noise and other environmental factors are also considered and kept at appropriate levels. Safety measures which protect personnel and property from injury or illness include the following: fume hoods, fire extinguishers and blankets, alarm systems, safety training, protective clothing, emergency showers, eyewashes and spill control kits.

Accreditations, Certifications, Licenses and Registrations

Triangle Laboratories of RTP, Inc. has received approval from several state and national agencies. The American Association for Laboratory Accreditation has conferred upon Triangle Labs accreditation for technical competence in environmental testing. The laboratory has been validated by the United States Army Corp of Engineers, and while not currently under contract, Triangle Labs has performed organic analyses under the United States Environmental Protection Agency (USEPA) Contract Laboratory Program. We are registered under current Food and Drug Administration (FDA) regulations to engage in the testing of drugs. We have received registration under the provisions of the Clinical Laboratory Improvement Amendments of 1988 (CLIA) to perform high complexity testing (dioxin and PCB's) of human samples. Triangle Labs has been licensed, under state regulations, by the North Carolina Department of Environment, Health, and Natural Resources to receive, possess, transfer, and import radioactive materials for the purpose of chemical analysis. We have been provisionally certified by several USEPA regions to analyze drinking water samples for dioxin. Many state agencies that have presented certifications to Triangle Labs can be found in Appendix 1B.

Section 6

ORGANIZATION AND PERSONNEL

Organization

Responsibility and Authority

At Triangle Laboratories, the management structure is best illustrated by referring to the Organizational Chart in Appendix 1A. Responsibilities and authority of key personnel found on the charts will be summarized later in this section. Brief resumes of key Triangle Labs personnel may be found in the company's Statement of Qualifications.

Verification Resources and Personnel

Verification activities include inspection and monitoring of process and product quality and auditing of the quality system, processes and products. Provision is made for personnel to be trained and have responsibility for these activities. Production personnel, under the direct supervision of Work Group Leaders, are responsible for the inspection and monitoring of in-process and final products. Audits of the quality system and products are performed by personnel independent of those having direct responsibility for the work being performed. Quality system audits are carried out by Quality Assurance Unit (QAU) personnel, while data audits (audits of the final product) are carried out by employees in both Client Services and the QA Unit.

Effective verification activities are achieved by the provision of adequate resources to personnel. These resources include adequate training, time for verification activities, knowledge about requirements, documented procedures, access to quality records, and adequate supplies and equipment necessary to perform verification.

Management Representative for Quality Assurance

The Quality Assurance Manager reports directly to the President, functions independently of production, and has the authority to implement and maintain the quality system. The management of Triangle Labs presents a strong commitment towards the important role of quality assurance in its organization. The Quality Assurance Manager and QAU personnel interact frequently with personnel at all levels throughout the organization.

Management Review

A formal management review of the quality system occurs at a minimum of once each year. The purpose of this review is to ensure that the quality system remains effective, meets the quality objectives and policies stated in Section 4 of this manual, and satisfies the requirements of state, national, and international certifications held by Triangle Labs. Records of management reviews shall be maintained in the QA Unit.

Personnel

Job Descriptions of Key Technical Personnel

While not all-inclusive of assigned duties, the following are brief descriptions of the chief technical personnel at Triangle Labs.

President/Chief Executive Officer: management of administrative, business, quality assurance, personnel and production activities, through direct supervision of the General Manager and the Quality Assurance Manager; minimum qualifications - education: Ph.D. Chemistry, experience: 10 years analytical chemistry.

General Manager: management of administrative, business, personnel and production activities through direct supervision of the Vice President of Administrative Operations, the Production Manager, the Systems and Internal Sales Manager; and personnel in the departments of Client Services, Personnel, and Safety and Health; minimum qualifications - education: B.S./B.A. in a management related field or equivalent, experience: 5 years general management.

Quality Assurance Manager: coordination and management of the Quality Assurance Unit; reports directly to the President; responsible for overseeing all quality aspects of the laboratory; specific elements to be maintained are: the laboratory Standard Operating Procedures, Quality Assurance Program and Quality Assurance Manual; coordination of internal and external audits, performance samples and laboratory certification data; minimum qualifications - education: B.S. Chemistry or equivalent, experience: 3 years in scientific field.

Production Manager: management of production operations, including supervision of work group leaders for the extraction lab, extraction lab support, GC/MS, GC, inorganics, report preparation and engineering groups; minimum qualifications - education: B.S. Physical Science, experience: 5 years general analytical chemistry, 2 years supervisory.

Recruitment Policy

The Personnel Department of Triangle Labs uses several methods of recruitment. Current employees are offered the earliest opportunity to apply for openings within the facility by posting available positions on the bulletin boards one week before outside sources are considered for candidates. Then, announcements are made in local newspapers, placement agencies (temporary and permanent), colleges and the Employment Security Commission offices. The recruitment process consists of collecting applications and resumes, distributing them to the appropriate supervisors, scheduling interviews as requested by supervisors and having candidates meet with relevant staff and a representative from the Personnel Department. The references of promising candidates are investigated prior to making job offers.

Training

Training is provided for new employees and as continuing education for veteran employees, both at Triangle Labs facilities and off-site.

On-Site Training: Training goes on at different levels throughout the facilities. Numerous manuals, texts, videos, SOP's, journals, analytical protocols and in-house instructors are available to trainees. On-the-job training related directly to the position is done by WGL's or other qualified staff. Typically, a trainee goes through a stepwise method to learn procedures pertaining to such areas as analytical methodology, report generation or quality assurance activities: he is given an SOP to read, he observes the trainer performing the procedure, he assists the trainer in performing the procedure several times, he performs the procedure without assistance but with the trainer's frequent inspection of his work, and finally, he may perform the procedure without supervision. A written QA Program is provided to all employees whose activities have a direct impact on product quality. Cross training, supervisory training and other related training takes place on a scheduled basis and is documented for training files.

Off-Site Training: This type of training takes place on an as-needed basis. Recommendations and suggestions about promising educational programs come from all levels of staff. Completed studies are documented and updated regularly in the training files. Courses may be taken at local colleges and universities. Workshops and seminars are often made available by instrument manufacturers, software companies and national associations specializing in analytical chemistry or laboratory quality assurance.

Records Maintenance

Résumés, education and experience records, job descriptions and training records are maintained by the personnel department: Résumés are put in a uniform format upon hire. These résumés are updated on an annual basis or as needed. Additional education and experience is updated with the résumés. There is a job description for each position existing within the company. Active training records are kept on file in the Quality Assurance Unit. All new and ongoing training information is periodically turned in by the area supervisors and the records updated at that time. These files contain records for any pertinent on- or off-site educational experiences, orientation records, SOP competence records or self help courses, such as "Smoke Stoppers" or Stress Management.

Safety and Health Policies

All personnel undertake a one day orientation upon initial employment and on-the-job intensive training concerning health and safety issues. Triangle Labs complies with the OSHA requirement that safety and health training takes place on an annual basis, with a careful introduction to new principles. We have contracted with Duke University Medical Center Occupational Health Services to provide us with recommendations for the improvement of the safety and health practices at Triangle Labs and periodic medical examinations for all employees. Triangle Labs' policy with respect to health and safety issues is presented in detail in several documents, with which employees are provided.

Section 7

ANALYTICAL SERVICES

Triangle Labs is a full service environmental analytical laboratory. Services provided include the preparation and analysis of a wide variety of sample matrices for such analytical categories as: Volatile and Semivolatile Organic Compounds, including Polychlorinated Biphenyls, by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry; Pesticides and Herbicides by High Resolution Gas Chromatography; Polychlorinated Dibenzo-*p*-Dioxins, Polychlorodibenzofurans, Polybrominated Dibenzo-*p*-Dioxins, Polybromodibenzofurans, Polychlorinated Biphenyls, and Polynuclear Aromatic Hydrocarbons by High Resolution Gas Chromatography/ High Resolution Mass Spectrometry; Polychlorinated Dibenzo-*p*-Dioxins and Polychlorodibenzofurans by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry; Adsorbable Organic Halides and Total Organic Halides by Adsorption and Microcoulometric Titration; and Inorganics by Ion Chromatography, Atomic Absorption Spectrophotometry, and Inductively Coupled Plasma-Atomic Emission Spectrophotometry. Triangle Labs is experienced in the analysis of many matrices, including air, aqueous, plant and animal tissues, soils, and other solids. Air matrices currently analyzed include Modified Method 5 (MM5) samples and Volatile Organic Sampling Trains (VOST). Several auxiliary services are also offered, such as the provision and preparation of sampling containers (e.g., XAD traps, VOST tubes, and bottles).

Analytical Methodology and Target Compounds

Triangle Labs utilizes a variety of published and in-house analytical methods. Minor modifications of methodology may be employed in some cases. Such modifications are validated prior to implementation in the laboratory. Target Compound Lists (TCL's) are chosen from the analytical methods. Published methodology utilized for each category of analytical services is listed below. Additional information about analytical services and methodology can be found elsewhere in this manual, in the Triangle Labs Price Book, and in several Data User Manuals. Selected analytical methods are summarized in Appendices 2 and 3 of this manual.:

Volatile Organic Compounds (VOA) - Methods 8240, 624 and 1624;

Semivolatile Organic Compounds (SVOA) - Methods 8270, 625 and 1653;

Pesticides - Methods 8080 and 608;

Herbicides - Method 8150;

Polychlorinated Biphenyls (PCB's) - Modified Method 680;

Polychlorinated Dibenzo-*p*-Dioxins (PCDD's) and Polychlorodibenzofurans (PCDF's) - Methods 8290, 23, 1613, 8280, 613 and NCASI 551;

Adsorbable Organic Halides (AOX)/Total Organic Halides (TOX) - Methods DIN 38409, DIN 38414, EPA 9020, EPA 1650, PTS-RH: 012/90, SCAN-W 9:89, ISO/DIS 9562, and APHA 5320B;

Inorganics - Ion Chromatography by Methods 7D, 26, 26A, 218.6, 300.0, and 9057; Trace Metals analyses by Methods 200.7, 6010, 7020, 7040, 7041, 7060, 7080, 7091, 7131, 7140, 7200, 7210, 7380, 7420, 7421, 7450, 7460, 7470, 7471, 7481, 7520, 7610, 7740, 7760, 7770, 7840, 7841, and 7870;

Triangle Labs offers analyses for Polynuclear Aromatic Hydrocarbons (PAH's), Polychlorinated Biphenyls (PCB's), Polybrominated Dibenzo-*p*-Dioxins (PBDD's) and Polybromodibenzofurans (PBDF's) by High Resolution Gas Chromatography/High Resolution Mass Spectrometry, employing methods developed in-house that utilize state-of-the-art technologies. Several protocols for PBDD/PBDF analyses, developed by Triangle Laboratories and approved by the EPA, follow the Testing and Reporting Requirements, Final Rule from 40 CFR, Part 766 of the Federal Register.

Contract Review

For all analytical services to be provided by Triangle Labs, contract review is accomplished through the generation of a written quote or contract. A written quote is utilized for short-term contracts, usually consisting of one analytical project. A written contract is utilized for long-term contracts consisting of multiple analytical projects. Sales and Client Services personnel are responsible for implementing and documenting contract review. Client requirements, including special needs that are not normally provided by Triangle Labs, are defined and documented on the written quote or contract. It must be determined whether special requirements can be met. Client Services Managers, who each have expertise in specific analytical services, are consulted in order to make this determination. If it is decided that the special requirements cannot be met, this is discussed with the client, and a counterproposal may be offered. Sales and Client Services personnel keep informed about the capacity of the lab to fulfill the different analytical services, in order to ensure that contractual requirements can be met.

Subcontracted Analyses

In dealing with any analyses that Triangle Labs cannot perform, there are established procedures for subcontracting. Two courses of action may be followed, depending on the nature of the client's requests for analyses. The client may be referred directly to another laboratory, if known; or work may be subcontracted by us to another laboratory. The latter usually takes place when Triangle Labs is able to perform part of the requested analyses. When a subcontracted analysis is one for which Triangle Labs is certified to perform, it must be determined that the subcontract lab has a quality assurance system in place that is consistent with our own. Incoming samples to be subcontracted are subjected to normal sample receipt procedures by the sample custodian. They are prepared and shipped to the subcontract laboratory. Results are received at Triangle Labs, a copy is sent to the client, and the original is archived. Triangle Labs invoices the client for the subcontracted work.

Section 8

LABORATORY MATERIALS—PURCHASING AND HANDLING

Purchasing, Receiving, Inspection, Inventory and Storage of Laboratory Materials

Practices utilized for the requisition, purchase, receipt, inspection, inventory, and storage of laboratory materials are described in several SOP's. A completed purchase requisition form provides a clear description of the product ordered. This includes, where applicable, a precise identification and reference to any specifications that must be met. Purchases are pre-approved by department heads. The purchasing department orders the material, from an approved supplier whenever possible. Upon receipt of the goods, receiving personnel examine them for damage before signing the bill of lading. Within two days, items and quantities in all shipments are compared with what was ordered and this information is communicated to purchasing and accounts payable. All stocked items are stored in the warehouse and a monthly inventory is performed. Non-stocked inventory is forwarded to the requisitioning person. Reagent materials are assigned expiration dates and placed on shelves so that the older materials will be used first.

Sample Container Cleaning, Storage, Preparation and Shipping

Triangle Labs does not perform sampling, but sampling kits may be provided should clients request sampling materials. All vials, jars, and bottles contained in the kits are purchased and must be QC class, precleaned, with a certificate of test results provided for our files. Glass sample containers are wrapped in two sheets of bubble wrap. The containers are placed in a plastic cooler with non-frozen ice packs, along with Chain-of-Custody forms, seals and labels enclosed in a ziplock bag. The kit is filled with styrofoam chips and sealed with tape for shipping. Since kits are assembled only upon clients' requests, no "ready for shipping" kits are stored. Precleaned glassware is stored in small quantities in house. Sampling materials, such as XAD traps, PUFs and VOST tubes, are also provided to or owned by the client. These are prepared, stored and handled as detailed in several SOP's. Preparation for shipping is the same as for empty glass bottles.

Glassware Cleaning

All glassware used in the High and Low Resolution wet labs is cleaned as described in written procedures. These procedures include solvent pre-rinses and soapy water washes. Basins and brushes are kept segregated so that cross contamination is kept to a minimum. High resolution glassware is subjected to a solvent soak and rinses with several different solvents. Low resolution glassware receives tap water rinses and is air dried and baked. All clean glassware is covered with aluminum foil and transferred to their proper locations, taking care that the glassware is not mixed up. In the Inorganic area, glassware is cleaned by a washing procedure that exceeds EPA guidelines. The glassware is washed with detergent, followed by acid soaks and multiple rinses with deionized water. The clean glassware is air-dried and stored in plastic bags.

Section 9

ANALYTICAL STANDARDS

During the analytical process, it is possible to obtain a variety of measurements. These include such measurements as volume, weight, concentration, pH, and temperature, to name just a few. The laboratory must implement practices that facilitate the traceability of these measurements to recognized standards of measurement.

Chemical Standards

The procurement, preparation, handling and storage of chemical standards is critical to the analytical process. It is through these chemical standards that reported analyte measurements in samples are traceable to reference values. Only the highest quality chemicals will be used as reference materials at Triangle Labs. Whenever possible, standard solutions will be traceable to national standards, such as NIST, EPA or A2LA certified reference materials. Numerous written procedures describe the management of these analytical standards. Procedures are written to ensure consistency with the requirements of analytical methods and current certifications and accreditations.

Sources of Standards, Traceability and Verification

Triangle Laboratories purchases standards from approved suppliers of chemical standards. Occasionally, clients supply standards specifically to be used in the preparation and analysis of their samples. Prior to using these standards, an agreement must be reached with the client about the handling and disposition of their standards. Information about these standards and any client requirements are recorded in the pertinent standards logbook. The chemist receiving a chemical standard shipment verifies that the information on the standard label is consistent with that on the supplier paperwork. Information about the standard is recorded in a standards logbook. Traceability of standard solutions is facilitated by the use of codes that unambiguously identify the supplier materials and all derived preparations. Standard materials of questionable composition or concentration may be verified against certified reference standards, when the latter are available.

Types of Standards

Analytical methodologies define a variety of standard solutions to be used by the laboratory. Among them are included: surrogate spikes, matrix spikes, internal standards, QC check standards, recovery standards, and calibration solutions. The composition and concentration of these solutions must conform to method specifications.

Standards are categorized at Triangle Labs according to the following definitions:

Primary Standard	A neat standard received from a supplier.
Stock Standard	A solution of a primary standard at a high concentration, used to prepare secondary standards. These may be prepared in-house or received from a supplier.
Secondary Standard	A solution of one or more stock standards, with each analyte prepared at a selected concentration, to be used as a beginning mixture for preparation of calibration or spike solutions. These may be prepared in-house or received from a supplier.
Working Standard	A solution which has been prepared in-house from secondary standard(s), or purchased from supplier, that will be used without dilution for instrument calibration or sample fortification.

Preparation of Standards

The preparation of any standard solution is performed by an experienced chemist (usually the Standards Preparation Chemist), and is documented in the appropriate standards logbook. New standard solutions are prepared as needed. The manner of preparation for a standard solution depends upon the required amount and concentration and its intended application. Several SOP's (in categories ODS, OAO, IIC, and INO) are utilized to assure the correct preparation and documentation of standard solutions.

All standards are assigned an expiration date. The supplier's assigned expiration date, if provided, is used for neat or primary standards. Otherwise, the expiration date is assigned based upon the supplier's date of preparation and the known stability of the analyte. (Some analytes are known to be highly volatile or to easily degrade or react.) When applicable, assigned expiration dates meet the requirements of analytical methods. A standard mixture is assigned an expiration date no later than that of the oldest components. The expiration date is only a guideline. Standards are frequently examined for deterioration and evaluated for their contribution to analytical problems. Standard solutions presenting signs of deterioration or for which the integrity can no longer be assured are replaced immediately.

Analyte or standard components common to calibration solutions and associated sample fortification solutions may be of the same primary source or an independent source. Some methodologies require that primary standards of the same supplier batch or lot number be used for both. Certain spiked QC samples must be prepared with reference material that is independent of the associated calibration standards. New standards are prepared as necessary to meet these requirements.

Inventory and Storage

Documentation for all standards is carefully recorded in relevant standards logbooks and/or a computer inventory system. The manner of storage for a standard is determined by its type and expiration date or shelf life. All organic and light sensitive standards are stored in amber vials or bottles, and are kept in designated refrigerators/freezers. Analytical standards are never stored together with samples and extracts.

Measurement Equipment

Equipment chosen for inspection, measuring, and testing shall meet all specific requirements for measurement capability identified by the pertinent analytical methods and certification agencies. This includes small equipment, such as thermometers, analytical balances, pH meters, autopipetors, and volumetric glassware; and large equipment, such as gas chromatographs and spectrometers.

Written procedures for the operation of measurement equipment, large or small, shall contain the information described below, where applicable. In addition, Section 11 on "Instrumental Analysis" contains more specific information about the calibration and operation of large measurement equipment.

- what equipment the procedure is to be performed on, including equipment type, identification number, and location;
- how the equipment is to be calibrated and used for measurement;
- what measurements are to be made;
- acceptance criteria for the calibrations, including the accuracy and precision required;
- corrective action for failed acceptance criteria, including assessment of previous calibration results;
- the basis used for calibration (e.g., national standards of measurement, such as NIST, ASTM, and A2LA; participation in EPA and state performance evaluations; round-robin studies with other laboratories);
- frequency at which the equipment will be calibrated, adjusted and checked;
- what records will be maintained to document the calibration and use of measurement equipment;
- how the calibration status for equipment is determined (e.g., a sticker or logbook entry);

- what environmental conditions are necessary before measurement equipment may be calibrated or used for measurement;
- what adjustments to measurement equipment, including software, cannot be made due to possible invalidation of the calibration setting;
- how measurement equipment is to be handled, preserved, and stored in order to maintain accuracy and fitness for use;

Section 10

SAMPLE RECEIPT, HANDLING AND PREPARATION

Sample Receipt and Chain-of-Custody

The Sample Custodian or a designated assistant will receive deliveries of all samples. A unique project number is assigned to each shipment of samples received from a client, and the first in-house records for the new project, including an internal Chain-of-Custody, are initiated. When samples are hand delivered by a customer, his name is recorded on the internal Chain-of-Custody. The shipping containers, their contents, and accompanying client documentation are examined by the Sample Custodian. Noted on the internal Chain-of-Custody is information about the presence and condition of custody seals and the state of preservation of the samples. Any discrepancies in documentation or problems with sample condition are also noted and brought to the attention of the client, who may provide clarification or further instructions. The Sample Custodian assigns an internal sample ID to each sample, which is labelled on the sample container. The following information pertinent to each sample is recorded on the internal Chain-of-Custody: internal sample ID, client sample ID, sample matrix and storage location. The original internal Chain-of-Custody is placed in storage with the samples. Sample Custodian SOP's (SMC category) describe procedures for sample receipt and log-in, chain-of custody, along with those for handling sample shipment containers provided by clients.

Sample Preservation and Security

Samples are stored in a manner which ensures their integrity and security. Samples are stored at temperatures which meet specifications of the methodology and client, in either a freezer at approximately -20° C, a refrigerator or cooler at approximately 4° C, or in a cabinet at room temperature. For most methods employed at Triangle Labs, required preservation techniques may be found in Appendix 5A. Quality Assurance Project Plans (QAPP's) often give specific preservation requirements that must be observed. Chemical preservative additions to sample containers normally takes place in the field at the time of sampling. Sample storage facilities at Triangle Labs are located beyond the punch lock security doors. Internal chain-of-custody procedures and documentation pertaining to sample possession, removal from storage and transfer are outlined in written procedures. Care is taken to ensure that cross-contamination does not occur during sample storage. Temperatures of cold storage areas are monitored and recorded twice daily, and corrective action taken as necessary. Walk-in coolers are monitored electronically 24 hours a day. Further details about sample storage and preservation may be found in the Sample Custodian (SMC) SOP's.

Sample Preparation Procedures

Samples are prepared in a way that is method and matrix specific. Most samples must be prepared within a method-specified time after sampling. These preparation holding times are complied with to the extent possible. Samples are occasionally received near or beyond the expiration of these holding times. For most methods employed at Triangle Labs, holding times may be found in Appendix 5A. Applicable Quality Assurance Project Plans (QAPP's) must be consulted for project-specific holding time requirements. Many primary extracts have to be subjected to clean-up procedures before they may be injected into a GC or GC/MS analytical system. All sample preparation procedures employed at Triangle Labs are covered by appropriate SOP's.

Sample, Extract, and Digestate Archival and Disposal

The Sample Custodian and other authorized personnel are responsible for the archival and disposal of raw samples, extracts, and digestates. Raw and prepared samples are not to be archived or disposed of until their designated analyses are complete and resultant analytical data are sent to clients. Samples in cold storage are retained there until at least 30 days after receipt. Archive samples are placed in boxes, labelled with the project numbers, and retained in a secured sample archive area for a specific length of time, prior to disposal. Written procedures describe routine archival and disposal practices. Clients are informed about these procedures and are given an opportunity to request exceptions to these routine practices. There is a storage fee for the retention of samples in cold storage or archive longer than the time established by routine practices.

Sample Return to the Client

When a client has requested the return of samples, the Sample Custodian prepares and ships the samples according written procedures. Protection of the samples during delivery is ensured by the implementation of special packaging procedures. Packages are delivered by a commercial carrier whose procedures for protecting the samples are not within the control of Triangle Labs. Clients are made aware that a commercial carrier will deliver their samples.

Sample Loss, Damage, or Unsuitability

It is possible for samples or sample containers to be lost, damaged or determined to be unsuitable, for whatever reason, after initial receipt at Triangle Labs. Whenever this happens, the event is recorded in the sample handling documentation by the observer. The problem is brought to the attention of a Client Services Manager, who will report it to the client. Plans for disposition of the affected sample(s) or containers are agreed upon with the client, carried out, and recorded in the project records.

Section 11

INSTRUMENTAL ANALYSIS

Instrumental analysis consists of setting up proper instrument operating conditions, executing acceptable calibrations and other instrument performance tests, analyzing prepared samples, and collecting data from the analyses. Instrumental analysis procedures, frequencies and acceptance criteria are described in several SOP's, the contents of which are derived from the source methods. A description of data collection and reduction at Triangle Labs is given in Section 12.

Instrument Operating Conditions

The published analytical methods normally define the optimum instrument operating conditions (e.g., temperature programs, column conditions, flow rates). Where applicable, these specifications will be followed, unless otherwise indicated for a project.

Calibration Procedures and Frequencies

The instrumental performance requirements of the published methods will be followed unless otherwise specified for a project. For all GC/MS methods, tuning and calibration (initial or continuing) is performed every 12 hours or less. The frequency of calibrations for other instrumentation is method specific. Other performance tests may also be executed to further demonstrate proper functioning of instrumentation. Calibration procedures and frequencies specific to instrument types are briefly described below.

Gas Chromatography/Mass Spectrometry (GC/MS)

Tuning and Mass Calibration

The high resolution mass spectrometer is tuned to give the required static resolving power, which is checked by using an oscilloscope. This measurement is confirmed by the use of a data system. The instrument is then mass calibrated using perfluorokerosene (PFK). Mass calibration is adjusted automatically to within +/- 5 parts-per-million approximately once per second during the course of all quantitative analyses.

The mass calibration of a quadrupole mass spectrometer is checked daily through the use of perfluorotributylamine reference compound (FC-43). The instrument is adjusted to give specified peak ratios for this compound, consistent with the type of analysis to be performed. The GC/MS is hardware tuned prior to performing the initial and continuing calibrations. Results must meet the peak ratio specifications of the analytical methods. For volatiles analyses, 50 ng of bromofluorobenzene (BFB) is used, and for semivolatiles analyses, 50 ng of decafluorotriphenylphosphine (DFTPP) is used.

Initial Calibration

The mass spectrometer response is typically calibrated by analyzing a set of five or more initial calibration solutions, as appropriate for each GC/MS method. Each solution is analyzed once. The relative response factor for each analyte (target compounds, surrog /internal/alternate standards) is calculated using the expression in Figure .1-1. The mean relative response factor for each analyte is then obtained using the expression in Figure 11-2. Integrated ion currents are utilized for these expressions. An acceptable calibration must meet the method specified criteria for percent relative standard deviations (%RSD) of the mean relative response factors, calculated for each analyte. Failure to me the criteria will result in corrective action (e.g., lo ating the source of the p blem and adjusting the instrument tuning parameters) before repeating the rejected analysis. Triangle Labs will not analyze any samples unless the performance criteria for calibrations are satisfied.

Continuing Calibration

A continuing calibration is demonstrated every 12 hours by injecting a solution with a concentration at or near the midpoint of the initial calibration range. The relative response factors for all analytes of interest are calculated and verified against the initial calibration mean relative response factors. The percent difference (%D) for each analyte is calculated using the expression in Figure 11-3. An acceptable continuing calibration run must have measured percent differences for the analytes within method specified ranges. Should any criteria for an acceptable calibration not be met, a new initial calibration will be established before any samples can be analyzed.

Figure 11-1

$$RRF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

RRF = *the relative response factor for the analyte*
A_{is} = *integrated area or ion current of the internal standard*
A_s = *integrated area or ion current of the analyte*
C_{is} = *amount of the internal standard*
C_s = *amount of the analyte*

Figure 11-2

$$\overline{RRF} = \frac{1}{n} \sum_{i}^{n} \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

$\overline{RRF}$ = *the mean value of the relative response factors for the analyte*
n = *the total number of data points derived from the initial calibration*
A_s, A_{is}, C_s, *and* C_{is} *have the same meaning as in Figure* 11-1

Figure 11-3

$$\%D = \frac{\overline{RRF} - RRF_{cc}}{\overline{RRF}} \times 100$$

where:

$\overline{RRF}$ = *the mean value of the relative response factors for the analyte from the initial calibration*

RRF_{cc} = *the continuing calibration RRF of the analyte*

Gas Chromatography/Electron Capture Detector (GC/ECD)

Initial Calibration

Internal standard calibration is utilized for the analysis of pesticides/PCBs and herbicides by GC/ECD. The method-specified number of calibration standards will be used. Each solution is analyzed once and the analyte relative response factors are calculated using the expression in Figure 11-1. The mean relative response factor for each analyte is then obtained by using the expression in Figure 11-2. Integrated areas are utilized for these expressions. For multiple response pesticides/PCB's, quantitation will consist of an average of the quantitated values for five selected peaks, if possible. The percent relative standard deviation (%RSD) must be less than ±20% in order to use the mean relative response factor for quantitation. If it is greater than ±20%, one more attempt is made to meet criteria. If the second attempt is unsuccessful, the analyst takes corrective action, such as instrument maintenance, and begins the sequence again.

Continuing Calibration

An initial calibration is verified through the performance of continuing calibrations at regular intervals throughout subsequent analyses. The frequency of continuing calibrations is method specific. The relative response factors for all analytes of interest are calculated and verified against the mean relative response factors from the initial calibration. The percent difference for each analyte is calculated using the expression in Figure 11-3. Relative response factors from the continuing calibration must be within 15% of the mean relative response factors. If any are not, another attempt is made to achieve an acceptable continuing calibration. If the second attempt is unsuccessful, corrective action is performed on the instrument and a new initial calibration is established.

Atomic Absorption Spectrophotometry (AA)

An initial calibration is performed daily with freshly prepared working standards. A four-point calibration curve is acquired which must have a correlation coefficient of 0.995 or better. The initial calibration is verified every 10 samples or 2 hours, whichever is more frequent. The continuing calibration is required to be within 10% or 20%, depending on the analytical method utilized. Continuing calibration blanks are run at the same frequency. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within 10% of the true value.

Inductively Coupled Plasma Emission Spectrophotometry (ICP)

Initial calibration is performed every 8 hours and continuing calibrations are performed every 10 samples or 2 hours, whichever is more frequent. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within 10% of the true value. The continuing calibration is required to meet the criteria of the analytical method.

Ion Chromatography (IC)

The ion chromatograph is typically calibrated by analyzing a set of five or more initial calibration solutions, with concentrations of analytes appropriate to the analytical methods. Procedures for verifying the calibration curve are method specific.

AOX/TOX Instrumentation

Instrumentation for the determination of AOX/TOX consists of a column adsorption module, titration cell and combustion/microcoulometric system. Several system performance tests are conducted and must meet acceptance criteria prior to sample analysis. The following performance tests are typically conducted, with slight variations between the different analytical methods. Granular activated carbon utilized in the column adsorption module is tested for purity. The titration cell is tested and adjusted based on the results of an injection of sodium chloride solution. Calibration of the combustion/microcoulometric system is accomplished through the analysis of method specific concentrations of 2,4,6-trichlorophenol. Verification of system performance and calibration is performed during sample testing according to specifications in the analytical methods.

Sample Analysis Procedures

Techniques for quantitative analysis of samples are specific to the analytical methods and sample matrices. Samples may either be subjected to a series of preparation steps prior to instrumental analysis, or they may be ready for analysis upon arrival at Triangle Labs. Most samples must be analyzed within a defined period of time following their collection, receipt at the lab and/or preparation. These analysis holding times are complied with to the extent possible (samples are occasionally received near or beyond the expiration date of holding time). For most methods employed at Triangle Labs, holding times may be found in Appendix 5A.

After sample analysis is completed and the data is processed, the analyst reviews the resultant data. If they do not meet established criteria, corrective action is taken to resolve problems. Once all the samples in a project have been analyzed and the data have met the criteria, the project documentation (instructions, raw data, reports, etc.) is sent to the next stage for preparation of the final report.

Section 12

DATA COLLECTION, REDUCTION, VALIDATION; REPORTING; DATA PACKAGE DELIVERY; AND SOFTWARE MANAGEMENT

Data Collection and Reduction

Quality assurance principles are applied in the acquisition of raw data related to chemical measurements. Raw data is that "primary data" which will be used to generate "secondary" data (the final analytical report). Raw data may be acquired manually or electronically. Manual data is hand written on data sheets and in logbooks. Electronically produced data is acquired from instrument and instrument/computer interface. Specific practices should be applied to each of these raw data forms.

Manual Data

Data sheets are standard, preprinted forms subject to document control. They may be bound into a book. Notebooks are bound, consecutively numbered, and subject to a controlled distribution and archival system. Manual data is entered as it is acquired, in permanent ink, signed and dated on each page should an entry take up more than one page. Corrections do not obscure any original entries and are made by cancelling with one line (no "white-out"), dating, initialing and, where appropriate, giving reasons for the cancellation in the margin. Unused portions of notebooks intentionally left blank are marked with a large X or Z.

Electronically Produced Data

Electronically produced data consist of chromatograms, spectra, data printouts, and raw quantitation reports (not the final report topsheets). Raw data for each sample and calibration are manually signed and dated by the responsible analysts, and must contain the full sample ID's or calibration name, file name, and date and time of acquisition. Any alterations to the raw data hardcopies and computer files is fully documented and clearly attributable to the person making such alterations (e.g., manual integrations are hard-copied for inclusion in the raw data, with area changes fully documented on the data printouts). There should be no ambiguity in data system printouts as to what peak on a chromatogram corresponds to an analyte of interest. Computer-collected data is reduced to hard copy as soon as possible, with a system of disk storage and backup disks to protect data and programs. Software used for data acquisition and quantitation reports is validated according to written procedures to assure that no "bugs" are present.

There are several different means of data collection, review and reduction, dependent upon specific methodology and instrumentation. Data review and reduction normally follow the guidelines of relevant EPA reference methods to the extent possible. For HRGC/HRMS analyses, established procedures consist of data acquisition and reduction on a Digital Micro VAX and

VAX 3100 and further reduction and data reporting using dBase software on a PC. For HRGC/LRMS analyses, established procedures consist of data acquisition and reduction using PC-based software or a PDP-11/24 system followed by further data reduction and reporting using dBase and/or spreadsheet software. For HRGC analyses, established procedures consist of data acquisition and reduction using PC-based software followed by further data reduction and reporting using dBase software. For AOX/TOX analyses, manual data acquisition from instrument panel readings is followed by data reduction and reporting using spreadsheet software.

All GC and GC/MS data go through several levels of review and inspection, starting with an initial QC examination in the Instrumentation area, followed by a thorough review in the Report Preparation/Data Review area. After preparation of a report, an independent review is performed by a Chemist other than the one who prepared the report. At each stage of the analytical process, data are reviewed for completeness, adherence to protocol requirements, and credibility. Results are fully validated, possible compromises of data quality are evaluated, and deviations from protocol requirements are documented. To the greatest extent possible, computer programs are utilized for data reduction. Where manual data manipulation procedures are required, data review is performed according to standard operating procedures. This ensures that the results are as independent of the Chemist performing the duties as possible. Corrective actions are implemented at the earliest possible opportunity.

All inorganics raw data are recorded on both paper printouts and on the instrument's computer disk drive. All analysis records are marked with the unique internal sample ID, the date, time, all replicate readings and dilution factors. All calibration data are also contained in these records. Data may be transferred to the report generation system by direct data transfer or, for small projects, manually transcribed.

Data Validation

These tests involve the performance of complex chemical analyses by a number of chemists. For this reason data validation and coordination are very important. At the conclusion of the analyses, the data are checked against the original shipping information and analytical request to be sure that the required analyses have been performed on all samples.

The validity of the data will be tested through the analysis of blank samples, duplicate samples and matrix spikes. The blank sample results will demonstrate the absence of laboratory contamination of the samples. Duplicate analyses give a measure of analytical precision. Analysis of matrix spike samples permits a measure of accuracy. Data for these QC samples are reviewed as soon as possible after analysis. For example, in the inorganics area, a data quality checklist is used by the instrument operator at the time of analysis, to verify that all calibration verifications are within tolerance, and that other QC indicators such as spike recoveries and blanks, are acceptable for a project.

Data Reporting

The data will be reported as components identified and the quantities present. The final report will include example calculations and descriptions of the equipment and procedures directly or by reference to a user manual. Complete data packages of all raw sample and calibration data will be prepared and archived. These will be furnished to the client upon request. Sample flagging procedures for HRGC/HRMS analyses are detailed in the published User Manuals for Dioxin, High Resolution PCB's and High Resolution PAH's. While no sample flagging is done directly on most HRGC/LRMS analytical reports, problematic results are discussed in the case narrative which accompanies each data package. Several standard report formats are used in the inorganics area, tailored to the data structure for the specific project type (e.g., TCLP, Multi-Metals Train or CLP).

Data Package Delivery

Data packages are prepared for delivery by the Shipping and Archive department according to their SOP's. Unless otherwise requested by the client, a copy of the data package is shipped, while the original is retained in a secured archive facility. The data packages are packed to meet the requirements of the commercial carrier chosen for delivery. Packages are delivered by a commercial carrier whose procedures for protecting the data packages are not within the control of Triangle Labs. Should the shipped data package be lost or damaged during delivery, a copy can be quickly prepared as a replacement. Clients are made aware that a commercial carrier will deliver their data packages.

Corrections and Additions to Documentation

The policy for handling additions/corrections of reports already issued is as follows. The Client Services Manager is to request an addition/correction in writing to the appropriate data review/report preparation personnel, who will make the requested change in a timely manner and internally verify the change. An authorized Chemist reviews and approves the addition/correction, and the Data Package Assembly Department mails or faxes the new report, which is then stored with the original data package for ten years.

Software Management

Triangle Labs has begun a formal validation program of its computer systems. Ultimately, the validation program is intended to be of a level such that all computer systems will meet the scope of any computer system audit. Our validation approach is three pronged. First, new software will be developed according to appropriate internal validation guidelines. Second, a validation committee has been appointed to oversee specific validation efforts of existing systems. Finally, systems will be kept validated through a system of change controls. This includes the *Computer Systems Services Request* (CSSR) forms which employees use to make known to the MIS department, desired changes to software and hardware. CSSR forms include personnel sign-offs for each step of the change process; and depending on the nature of the change, specify increasingly stringent required levels of authorization. Change controls also include software

version control; changes to existing software are announced, uniquely labelled, documented, and old versions are archived for future reference.

The goals of our software development methodology, existing system validations, and the change control system are to ensure that our software systems perform the required functions accurately, that the users understand how to use the system, and that auditors can assure themselves of the validity of our analytical methods. This in turn insures that we deliver accurate analyses to our customers in a timely fashion.

Section 13

DOCUMENTATION FOR QUALITY ASSURANCE

Objectives of Documentation

The objectives of documentation for quality assurance are to provide a standardized, written program of policies, procedures and instructions; and to prove that adequate quality assurance and quality control procedures are being implemented, accountability of the data is maintained, and traceability of analytical results is facilitated.

Document Control

The laboratory shall maintain control over the possession and distribution of all documents that directly impact on product or service quality. It is the responsibility of area supervisors (e.g., WGL's) to ensure that document control files are created and maintained for all applicable documents originating in their areas. This includes such documents as the Quality Assurance Manual, Standard Operating Procedures (SOP's), Quality Assurance Project Plans (QAPP's), data user manuals, client instructions, product sheets, and extraction flow charts. It includes standard forms, such as those for documenting sample chain-of-custody, tracking, extraction, clean-up, and observation; QC inspection checklists; system audit checklists; and corrective action reports.

A written procedure describes document control practices. Full or limited document control is applied, dependent upon the purpose of the document. Those publications which document the quality assurance system at Triangle Labs, specifically the QA Manual and Standard Operating Procedures, are subject to full document control practices. Limited document control procedures are employed for other relevant documents, such as forms, flow charts, data user manuals, and price lists. The procedure for limited document control allows for more than one revision of the same document to be active at the same time.

Every document is assigned a unique identification (usually a title, file ID and creation/revision date) which must be present on each page of the document. This unique identification is entered on a master list of documents, along with a distribution list for each document to ensure that pertinent documents are made available wherever they are essential. A master set of current documents is maintained along with the master list. Documents will be revised and re-issued after a practical number of changes have been made.

Full document control, as applied to the QA Manual and Standard Operating Procedures, also includes the following. The status of each document, active/current or inactive/obsolete is indicated on the master list. Each document and any subsequent revisions must be reviewed and approved by authorized personnel prior to issue. Personnel authorized to review and approve a document are to have access to all necessary information on which to base their review and

approval. Obsolete documents are to be retrieved from distribution points and replaced with current versions. The nature of changes in documents shall be identified within the document.

Standard Operating Procedures (SOP's)

Standard Operating Procedures (SOP's) are quality assurance documents in which instructions for every repetitive or standard operation performed by the laboratory must be detailed. There are hundreds of topics which must be covered by SOP's. The writer of an SOP should be the person most familiar with a topic. There is an SOP that describes the standard format for writing SOP's which will be of great assistance and will help the writer avoid overlooking anything. It is important that SOP's receive evaluation and input by laboratory supervisors and key technical personnel. The content of SOP's must conform to applicable requirements of analytical methods and certification agencies and be consistent with the Good Laboratory Practice standards. Within these constraints, the content of an SOP may be customized to meet the needs of a particular area of the laboratory. The performance of laboratory operations is subject to audit for compliance with written SOP's. If an SOP is impractical, hard to follow, or no longer meets laboratory needs, it must be modified or replaced.

The need for new or revised SOP's can be determined when a new method is implemented, when the scope of the existing method is extended or when some activities are being performed without adequate SOP's. Such a need can be identified by the analyst involved in the production or by someone from management. Also, the QA Unit may identify the need and request new or revised SOP's, usually as a corrective action for deficiencies found during an internal audit. SOP's are created to provide a clear, concise, step-by-step description of the procedure with explanatory information to enable a person with the appropriate background to perform the procedure. Revisions are made to SOP's at any given time to reflect changes in procedures. Each SOP is subject to review on its anniversary. A QAU SOP Coordinator notifies the appropriate person prior to the annual review date. If no revisions are necessary the next review date is established.

The QA Unit administers the Standard Operating Procedures program. Within the QA Unit, the SOP Coordinator is responsible for clerical preparation and distribution of new or revised SOPs, record keeping and archival of replaced and retired SOP's. Each SOP is assigned a unique identification, based on its functional category. Categories of SOP's are listed in Appendix 4A. The QAU SOP Coordinator distributes SOP's to appropriate area SOP coordinators and files the original approved hardcopy in the QAU Master Notebook. The area SOP coordinator is responsible for discarding copies of obsolete SOP's upon receipt of revisions.

Quality Assurance Project Plans (QAPP's) and Data User Manuals

QAPP's and Data User Manuals are documents that provide an overview of the way a project, a group of allied projects, or a specific analytical program is conducted. A QAPP is usually developed through the collaborative efforts of two or more companies. The general content and format of a QAPP is specified in *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans* (USEPA, December 1980), copies of which are available in the QA Unit. Data User Manuals are developed solely within Triangle Laboratories and are created to

provide essential information to clients, which aids them in understanding the data packages they receive. (Standard Operating Procedures are detailed laboratory instructions and are often proprietary. Proprietary procedures are not normally made available to clients.) QAPP's and data user manuals are prepared by Client Services Managers. Multiple versions may be concurrently in use. QAPP's and Data User Manuals are subject to limited document control.

Quality Records

Quality records must be maintained to prove that the quality assurance system is being effectively applied. At Triangle Labs, specific procedures for the identification, collection, indexing, filing, storage, maintenance, and disposition of various quality records are described in several SOP's. All quality records must be permanent (indelible ink), legible, attributable to those personnel who wrote them, and protected so they may not be adversely affected by an unsuitable environment. They are stored and maintained in a manner that facilitates rapid retrieval for a period of ten years after completion. Project specific quality records are available for evaluation by the client or his representative during the archive period of ten years. In fact, certain quality records, as specified by SOP or contract, are delivered to the client with the final product.

Project specific quality records are maintained to prove that adequate quality control procedures are being implemented, accountability of the project data is maintained, and traceability of analytical results is facilitated. Accountability means that reported data reflect the sample as it was received, that sample mix-up was avoided, and the sample was properly preserved after receipt. Traceability means that reported data may be reconstructed at a later date. Through proper documentation, a laboratory is able to demonstrate or prove to clients or government agencies that the quality of the data is what the laboratory says it is. Records must contain sufficient information to permit the reconstruction of calibrations, sample preparations and sample analyses.

Quality records that are maintained at Triangle Labs include, but are not limited to, the following.

- records for sample receipt, preparation and handling
- equipment/instrument calibration and maintenance records
- field sample and quality control sample analysis data
- project communication tracking forms
- inspection reports for receiving, in-process and final product
- subcontractor records
- vendor qualification records
- logbooks: run logs, maintenance logs, temperature logs, balance logs, etc.
- method validation records: MDL studies, initial precision and accuracy demonstrations
- control charts
- system and data audit reports
- corrective action reports
- QA reports to management

Many of these quality records are discussed at length in other sections of this manual. Laboratory notebooks (or "logbooks") are utilized throughout Triangle Labs for many different purposes. All logbooks are maintained according to a general written procedure, and other SOP's provide additional details for making entries in specific logbooks. New logbooks are issued by a system of signing them out in a designated logbook. Information that must be documented, both in the new logbook and the sign-out logbook, includes the assigned owner, the date issued, and the name and subject of the logbook. Logbooks must be maintained by initialing and dating every entry and z-ing down all areas of the logbook intentionally left blank. Many of the laboratory notebooks maintained at Triangle Labs are itemized in Appendix 6A. In addition to these logbooks, many personnel maintain personal logbooks, phone logbooks and pro :m logbooks. Bound logbooks are kept to document all monitoring, maintenance and calibration of analytical instrumentation, and such laboratory equipment as balances, refrigerators and ovens. Software and hardware records for computers are also kept in logbooks. Logbooks specific to equipment is kept nearby to ensure that the work is recorded concurrently. Upon completion, logbooks that contain quality records are stored in the Archive Room for a period of ten years.

Archive

The Archive Room is locked at all times and only appointed Archivists are allowed to possess keys to enter the Archive Room. All other authorized personnel may enter the room only in the presence of an Archivist and must sign and date a logbook in the Archive Room. Any materials removed from the Archive Room must be signed out by the Archivist.

All magnetic and hard copies of data, calibrations, equipment maintenance records, calculations, records of original observations, final test results and any other miscellaneous quality records directly associated with sample analyses are stored in a secured facility for ten (10) years after completion of a project. They may be stored in the Archive Room or at an off-site storage facility. Hardcopy records that are stored off-site are made available on-site in microfiche form. When completed or no longer in use, logbooks are also archived.

Section 14

QUALITY ASSURANCE

Through a formal quality assurance system, Triangle Laboratories of RTP, Inc. is able to prove that our product/service meets specific quality standards. These quality standards are defined according to the needs and requirements of our clients, the analytical methods utilized, government agencies, and senior management at Triangle Labs.

Quality assurance is a very broad and multifaceted concept, about which much confusion exists. It is composed of quality control and quality assessment. Quality control is the most important component of quality assurance. The need for quality assessment would be negligible if the laboratory always achieved perfect quality control.

> Quality control is a system of activities applied at each stage of the production process. Its purpose is to assure that products meet defined quality standards. This system includes the following: employee education, training, and experience; documentation (e.g., instructions, document control, records); instrument calibration and maintenance; laboratory accomodations; and inspection.
>
> Quality assessment is a system of activities employed to assure that quality control takes place at each stage of the production process. This system includes the following: system, data, and performance audits; reference materials; statistical evaluations; retests; and measurement bias investigation (when measurements may be operator-, instrument-, or methodology-dependent).

The success of a quality assurance system is dependent upon acknowledgement by all personnel of their responsibility for the system. Triangle Labs management is ultimately accountable for product quality, but no one person or group (e.g., the QA Unit) is responsible for the greater part of quality assurance program activities. Details of the program may be found throughout this QA manual. The remainder of Section 14 will be limited to a discussion of the Quality Assurance Unit (QAU), and the major activities performed and/or administered by this group.

The Quality Assurance Unit

At Triangle Labs, the QAU monitors the quality assurance system, as it is implemented throughout the laboratory, and reports the results of its observations to senior management. The Quality Assurance Manager reports directly to top management and the QAU has no direct responsibility for productivity in the laboratory. The objective of this independence is to eliminate all conflicts of interest in the performance of QAU duties. Major activities performed and/or administered by the QAU are summarized below. Each activity is discussed in greater detail elsewhere in the QA manual, as indicated.

- Performance of internal audits and coordination of external audits (see this section)
- Administration of a system for formal Corrective Action Reports (see this section and Section 15)
- Administration of laboratory certification/accreditation programs (see this section and Section 5)
- Performance of QAU duties required for GLP-regulated studies (see this section)
- Administration of the system for document control, with emphasis on the maintenance of Standard Operating Procedures (see Section 13)
- Performance of statistical evaluations for selected quality indicators, and maintenance of quality records (e.g., control charts, summary reports) generated to document selected statistical evaluations performed throughout the laboratory (see Section 15)
- Publication of the QA Manual and other documents that describe the quality assurance system at Triangle Labs (see Section 3)

Audits

There are several different types of audits. These may be internal, in which the laboratory reviews and examines itself, or external, in which the laboratory is audited by outside organizations, such as accrediting or regulatory agencies and clients.

Internal System Audits

A system audit is an on-site inspection and review of the qual assurance system as it is employed in the laboratory. During an audit, verification may be sought that: adequate written instructions are available for use; that analytical practices performed in the laboratory are consistent with SOP's; that adequate quality control practices are applied during production; that corrective actions are applied as necessary; that deviations from approved protocols are occurring only with proper authorization and documentation; that SOP's, quality records, analytical records, magnetic tape, etc., are properly maintained; and that personnel training records are satisfactory and current.

Internal system audits are implemented by the Quality Assurance Unit to assess the functioning of one or more department(s) of the laboratory. These evaluations are total audits of selected departments. Prior to a scheduled audit, checklists specific to a department's function are sent to the area supervisor. These checklists provide the department with many specific requirements and an opportunity to make immediate changes as necessary for compliance. The checklists are to be completed and returned to the QA Unit, where they are utilized to prepare for the audit. Since a department may utilize numerous SOP's and generate many records,

a comprehensive evaluation of all such documentation would be time prohibitive. Therefore, a representative sampling of departmental documentation is chosen for scrutiny during the system audit. Auditors interview the department supervisor and random employees, and observe work in progress.

After the audit, a formal System Audit Report is prepared by the QA Unit and sent to the area supervisor. The audit findings, of both compliance and non-compliance, are recorded and maintained as part of the QA documentation. This includes the audit checklists, the formal audit report, and any notes taken during the course of the audit.

After receiving a System Audit Report, the area supervisor must develop a written corrective action plan (including dates of implementation) for the deficiencies and recommendations cited in the report. This plan is submitted to the Quality Assurance Unit. The Quality Assurance Unit forwards copies of the audit report and corrective action plan to senior management for their review and approval. Timely corrective action must be made on the deficiencies. Deficiencies are expected to be corrected within three months after the date of the audit report, unless extenuating circumstances are detailed in writing to the QA Unit. QA Unit personnel follow up on corrective action implementation which may include modification of plans based on senior management reviews. System audit files remain open until corrective actions are completed.

System audit schedules are maintained in QA Unit files. Planned dates on the schedule are used as guidelines for sending audit checklists, performing audits, issuing audit reports, responding to audit reports, and following up on corrective actions.

External System Audits

Representatives sent by clients and government or accrediting agencies often perform system audits at Triangle Labs. These audits are most often announced inspections, but sometimes are conducted without forewarning. QA Unit personnel usually accompany such audit teams through the lab. The auditors receive a brief overview of company objectives, activities, and facilities. Interviews with essential supervisory and technical staff are arranged, along with retrieval of any documentation pertinent to the audit. Auditors typically provide an account of their findings shortly after the audit. This account is evaluated by QA personnel and reported to management, along with corrective action recommendations in response to any cited deficiencies.

Data Audits

Data audits are performed by technical personnel (in Client Services or the QA Unit) on a random sampling of the data reports produced at Triangle Labs. It is a goal to perform a comprehensive evaluation of approximately 10% of sample reports. The actual number is dependent upon available resources and the apparent effectiveness of QC inspections applied during production stages for particular types of analytical products. A data report is carefully evaluated for technical, clerical and administrative accuracy. Primary emphasis is placed on the ability of

the data report to meet customer requirements. Data audits are utilized for several purposes, including: identification of opportunities for process improvement, evaluation of the efficiency of the system, detection of inadequate execution of quality control procedures, early warning of potential system deficiencies, corrective action recommendations, and reports to upper level management.

Performance Audits

A performance audit is the analysis of a fortified blank sample, for the purpose of evaluating laboratory or analyst performance. There are several examples of performance audits, which may be of internal or external origin. Performance Evaluation (PE) samples have analyte concentrations unknown to Triangle Labs, and are submitted by external organizations. PE's may be analyzed as part of multi-laboratory round robin studies, in conjunction with accreditation programs, or as blind check samples submitted by clients. Internal performance audits are fortified blanks with known analyte concentrations, the values of which the analysts may or may not be aware. Examples of internal performance audits include initial precision and accuracy studies, blind check samples, laboratory control samples, and blind samples. The results of performance audits are utilized for several purposes other than the evaluation of laboratory performance, including: to fulfill accreditation requirements, to serve as analyst proficiency tests, and to facilitate laboratory improvement efforts.

Corrective Action Reports

All major or non-routine problems, deficiencies, or irregularities must be reported to management. A formal Corrective Action Report (CAR) system, administered by the QA Unit, is in place at Triangle Labs. The QA Unit issues CAR forms, monitors the progress of corrective actions, maintains completed documentation, and provides reports to senior management on the status of formal corrective action activities. A blank report form may be found in Appendix 7A. CAR's may be originated by anyone responsible for the quality of a product. A completed form is sent to an appropriate person or group to whom responsibility for corrective action is assigned. One person is designated the Corrective Action Analyst. This person records the corrective action plans, implementations and follow-ups as completed by the responsible person(s). During the corrective action process, several measures may be taken. These include: determination of the root cause through careful analysis of processes, specifications, quality records, customer complaints, etc., using statistical process control when applicable; implementation of measures that prevent recurrence of the problem; implementation of process controls to ensure that effective corrective action is taken; application of remedial actions to products affected by the identified problem; and revision of documentation for procedures that have undergone change as a result of corrective action.

Certification and Accreditation

Triangle Labs has been granted numerous certifications and accreditations, based upon compliance with standards set forth by the granting agencies. These credentials have enabled Triangle Labs to expand and retain a substantial client base. More information about specific credentials can be found in Section 5, page 3. The nature of the quality assurance program

implemented at Triangle Labs is profoundly affected by requirements of certification agencies. The QA Unit is responsible for the administration of certification programs. Several issues must be considered in the administration of these programs, including:

- Agencies may perform scheduled and surprise on-site audits
- Agencies may require acceptable analyses by the laboratory of performance evaluation (PE) samples
- Agencies require written corrective action responses to deficiencies cited in audit reports and performance evaluation results.
- Information about the requirements and status of certifications must be disseminated to relevant laboratory personnel.
- Agency requirements for quality assurance (e.g., QC samples, control charts) may exceed method requirements and may vary from one agency to another
- Administrative details of certification must be attended to (e.g., application, reapplication, filing, telephone and mail communications, fee payment, etc.)

GLP Regulated Studies

The Good Laboratory Practices (GLP's) are a set of regulations decreed by the United States Environmental Protection Agency (EPA) and the United States Food and Drug Administration (FDA), both of whom are concerned with the toxicology of chemical substances. Compliance with these GLP's is required for certain projects ("studies") completed at Triangle Labs. The GLP's define some specific responsibilities for the Quality Assurance Unit. Briefly summarized, these QAU duties include the following:

- Maintain a copy of the master schedule sheet for all studies
- Maintain copies of all protocols pertaining to all studies
- Inspect each study at adequate intervals
- Submit written status reports on each study to management and the study director
- Determine that no deviations from approved protocols or SOP's were made without proper authorization and documentation
- Review the final study report
- Prepare and sign a statement to be included with the final study report

Section 15

QUALITY CONTROL

At Triangle Labs, quality control is achieved through the application of a many practices. Quality control activities commence before production is initiated, and are assimilated at each stage of the production process. The purpose of these activities is to assure that all required standards of quality are met. Quality control activities are described in many sections of this manual. The remainder of this section will describe a subset of quality control activities that may be considered a discrete process, summarized as follows:

Prior to the initiation of production activities, required quality standards are defined. These are derived from several sources, including: requirements of the analytical methods, needs stated by the clients, and standards established within Triangle Labs.

During production, verification activities are performed to determine that defined quality standards have been met. Also, preventive measures are applied to avoid the possibility of nonconformity.

When defined quality standards have not been met (nonconformities), corrective actions are applied and verified to determine that the results meet requirements.

Data Quality Objectives

Data are produced for clients at Triangle Labs. Defined quality standards for these data may be expressed as data quality objectives (DQO's). These are established prior to sample preparation and analysis. Quality assurance indicators common to all DQO's include, but are not limited to: accuracy, precision, completeness, representativeness, and comparability. Examination of the QA indicators is performed to demonstrate that the data are scientifically valid, legally defensible and that they adequately meet established DQO's. The QA indicators may be summarized as follows:

Accuracy A quantitative measure of the relationship of reported data compared to the "true" or expected values. This measurement may be accomplished by evaluation of the recoveries of analytes spiked into samples. Specific accuracy measurement activities include surrogate spikes, matrix spikes and Quality Control Check Samples.

Precision A quantitative measure of the reproducibility of measurements made under controlled conditions. This measurement may be accomplished by comparison of recoveries of analytes in replicate samples or injections. These analytes may be spiked or native to the duplicate samples. Specific precision measurement activities include blind field replicates, lab replicates, matrix spike replicates and replicate injections.

Completeness A qualitative measure of the amount of valid data obtained from the analytical process compared to the amount that was expected to be obtained. Valid data must meet all data quality objectives for precision and accuracy.

Representativeness A qualitative measure of the degree to which data represents the characteristics of the population from which samples were collected. This is usually dependent upon sampling techniques not controlled by the analytical laboratory, however, there is representativeness of subsamples prepared within the laboratory from collected samples. Parent samples must be subjected to thorough homogenization prior to subsampling.

Comparability A qualitative measure of the confidence with which one set of data can be compared to another. Characteristics that make comparison possible include standardized report format, consistency of units (e.g., µg/L, ppm), and standardized sample preparation and analysis.

Quality Control Samples and Spikes

Analytical performance is monitored through quality control samples and spikes, such as laboratory method blanks, surrogate spikes, quality control check samples, matrix spikes, matrix spike duplicates, duplicate samples and duplicate injections. Many of these quality control measures, as applied at Triangle Labs, are summarized below.

Laboratory Method Blank A laboratory method blank consists of a sample that is processed in a manner identical to that of a regular sample, except that the matrix is replaced with distilled water for aqueous matrices, sodium sulfate for solid matrices, XAD-2 resin for MM-5 and PUF filter for PUF air sampling cartridges. The laboratory method blank sample is fortified and prepared along with the field samples, at a frequency of one laboratory method blank per batch of 20 (or less) field samples of a given matrix type. The laboratory method blank serves to demonstrate a contamination free environment in the laboratory.

Surrogate Standards For certain methods, all samples, including the laboratory method blank, are spiked with a set of specific surrogate standards to monitor accuracy of the analytical determination for each particular sample. QC criteria for surrogate recoveries are method and matrix specific. Laboratory QC criteria are established upon acquisition of a sufficient number of data points (20 or more), or else, the limits specified in each method are utilized.

Quality Control Check Sample A quality control check sample consists of HPLC grade water for aqueous matrices or precleaned sand for solid matrices. The QC check sample is fortified not only with appropriate internal and/or surrogate standards, but also with target analytes. QC check samples are analyzed at a frequency dependent on the method. They serve as an estimation of system precision and accuracy. Results of QC check samples are monitored on control charts, with QC requirements for recoveries being established as they are for surrogate recoveries.

Matrix Spike Sample

A matrix spike (MS) sample consists of a field sample, identified by the client, that is split into two parts and processed in a manner identical to that of the rest of the field samples. However, in addition to the regular fortification with the standards (internal, surrogate and/or alternate), the chemist will add a set of the target analytes to one part of the chosen sample before the preparation. The fortification levels for the target analytes are defined by the analytical method or the client's request. At the request of the client, one such sample will be prepared for every batch of 20 samples (or less) for a given matrix. To be able to run matrix spikes, the client must provide Triangle Labs with extra sample amounts.

The analytical report for the matrix spike will contain a tabulation of the analyte concentrations as expected and as measured, along with the calculated percent recoveries based on the expected concentrations. The percent recoveries actually represent a measurement of the method accuracy for that particular sample and matrix. Accuracies are established and updated for a particular analyte and method. In the absence of observable quantitative interferences, the MS sample showing accuracies falling outside the QC limits must be reanalyzed unless the matrix spike duplicate (MSD), which was processed along with the MS, shows similar deviations as a result of a "matrix effect." This type of corrective action can only be implemented if the sample selected for the MS (and MSD) was proven to be free of the target analytes, or did not contain high concentrations that significantly exceed the MS fortification level of these analytes. "Matrix effect" is further substantiated by acceptable recoveries in a QC check sample processed along with the field samples. Matrix spike recoveries, and the possible effects on data quality when accuracies fall outside the QC limits, are discussed in the Case Narrative.

Matrix Spike Duplicate Sample

The matrix spike duplicate (MSD) sample is commonly prepared (at the Client's request) in conjunction with the matrix spike (MS) sample. The analytical report will summarize the data from the MS and MSD analyses in a format allowing determination of the precision of the analyses. As for the matrix spike sample, the client must provide Triangle Labs with extra sample amounts.

Duplicate Sample

A duplicate sample (DUP) consists of a set of two samples obtained in an identical way, from the same field during exactly the same sampling session. The collection of duplicate samples from an inhomogenous matrix requires good planning and skilled technicians. At the client's request, one such sample per batch of 20 samples (or as specified by the client), per matrix type may be analyzed, provided the client supplies Triangle Laboratories with relevant samples.

The analytical report for the duplicate sample(s) will contain a tabulation of the results showing the precision as Relative Percent Difference (RPD). Precision exceeding any specified target values will necessitate a corrective action to assess the influence of the sampling procedure. The RPD is calculated as follows:

$$RPD = \frac{X_1 - X_2}{(X_1 + X_2) / 2} \times 100$$

where:
RPD = *the percent relative difference*
X_i $(i=1,2)$ = *the analyte concentration in the regular sample* (1), *and in the duplicate sample* (2).

Duplicate Injection

Upon client request, a duplicate injection of the same sample extract will be performed. In the absence of observable quantitative interferences, the RPD should be within ±30% or the two injections will be repeated after identification of the problem. Field samples analyzed during a suspected out-of-control situation will be reinjected as well.

Statistical Evaluation

Statistical evaluations can be made of selected analytical quality indicators, including spike recoveries, calibration responses, contamination levels, and method detection limits. Production units monitor levels of compliance with many criteria on a "real time" basis. Efforts are also being made to establish a system for "real time" statistical process control (SPC). Control charts offer the most graphic representation of a statistical evaluation. Control charts serve to identify the occurrence of a problem, but the cause must then be promptly investigated and eliminated. In-house QC criteria may be determined through historical trend analysis of data collected on QC charts. Selected statistical evaluations are performed by both the QAU and production units. A central file of documented statistical evaluations is maintained by the QAU.

QC Inspection

The main purpose of inspection is to determine the extent to which in-process and final products meet requirements. Written procedures (SOP's) for performing these inspections are available to employees responsible for such work. Documented product specifications (in SOP's, data user manuals, QAPP's, QC guidelists, published analytical methods, and specific project instructions) are also provided to production personnel to enable them to inspect for conformance. Specific quality records are created as work on a project progresses, beginning with client sample receipt and preparation, through instrument calibration, sample analysis, data review and report preparation. This documentation further enables product inspection to take place.

Certain requirements must be met for all inspections. Inspection records must be created to document the fact that inspection has taken place. The inspection and test status of the product is documented in these records to ensure that the product is not released until it conforms to all requirements. Detected nonconformances must be recorded during the inspection. Corrective action must be taken and documented whenever nonconformance is detected. The identity of the inspection authority responsible for releasing the product is documented in the inspection records. Until required inspections are performed on the intermediate and final product, it is not permitted to progress further along the production process, except by special client request.

In-process Inspection and Testing

Each work group is responsible for a segment of the production process and for all in-process inspection and testing that takes place within their work group. In-process inspection is accomplished through 100% screening for all work groups. Each client sample that goes through the analytical process is unique and can be considered a separate lot.

Final Inspection and Testing

The last stage of the production process is the preparation of a client-worthy data package and case narrative. This requires a thorough review of all records generated for a client sample since its receipt, including inspection records and any client documentation that may have originated before sample receipt. A Report Preparation Chemist performs this function during the preparation of the data package and case narrative. This inspection serves as both an in-process and final inspection of the product. In addition, a second chemist performs another final QC inspection of the data package and quality records. Again, any nonconformances found during these inspections must be corrected before the data package is released. Approval of the data package for release to the client is indicated by the signatures of the Report Preparation and QC Chemists on the case narrative.

Nonconformity

Each field sample that is incorporated into the analytical process is unique. Laboratory procedures are designed to introduce as much standardization as possible. Whenever conformance to standards is uncertain, the product is reviewed to determine the nature and cause of nonconformance. If it is judged to be nonconforming due to the unique nature of a sample, there may be little recourse other than to simply inform the client. It may be possible to utilize less standard procedures to provide a more useful product to the client.

Product nonconformance is identified either through inspection or coincidentally with work being performed on the product. Events of nonconformity are recorded in the quality records that accompany the work instructions for products. All employees are explicitly responsible for making themselves aware of these quality records before working on the product. Corrective action for nonconformities identified in the quality records must take place before work on the product is resumed.

Responsibility for the review and disposition of nonconforming products, what action is taken, and what functions are notified of the nonconformity depends upon the nature of the problem. For certain types of nonconformities, the procedure is defined in the SOP's. There are several ways that assignment of responsibility for review and disposition can be made. It may belong to the person who detected the nonconformity, the person who was working on the product, the Work Group Leader for the area where the nonconformity originated, a quality team, or personnel in the Client Services Department. The unique nature of every sample makes it impossible to specify what to do in every case. For those nonconformities where there is no precedent or documented procedure for review and disposition, responsibility lies with personnel in the Client Services Department. Personnel who have the authority to review specific nonconformities normally have the authority for deciding what will be done about it. Alternatives for disposition

include partial or complete rework, additional work agreed upon by the client, and acceptance by the client of nonconforming product. Rework is subject to the same inspection procedures as the initial work. Nonconformity, its review, and its disposition must be documented in the quality records as prescribed by the written procedures.

Corrective and Preventive Action

Appropriate actions must be taken to prevent or correct nonconformities in products and problems in analytical systems. When actions result in permanent procedural changes, pertinent documentation (e.g., SOP's) must also be modified to reflect these changes. Cost-effective preventive measures are applied whenever possible. In specific cases, the cost of applying preventive measures would exceed the cost of applying routine corrective actions. Because every client sample possesses unique and unknown properties, some predisposition to unpredictable, unpreventable nonconformities exists.

Corrective Action

Specific corrective actions are of two types: routine corrective actions applied to solve minor or commonplace problems, and formal corrective actions taken to eliminate major or non-routine problems.

Routine corrective actions are usually made by the chemists, technicians or instrument operators who detect minor problems or product nonconformances. SOP's that describe procedures for working on the product generally contain instructions for implementing and documenting corrective actions for typical problems.

There are three procedures for conducting formal corrective actions. The first is corrective action in response to a system audit report from the Quality Assurance Unit. This procedure is more thoroughly described in Section 14, page 3. The second procedure is the formal Corrective Action Report, which may be initiated by anyone who detects a significant quality problem. This procedure is also administered by the Quality Assurance Unit. Further information about it can be found in Section 14, page 4. The third practice is described in a written procedure on "Problem Sample Communication." It is initiated in response to client complaints about specific projects.

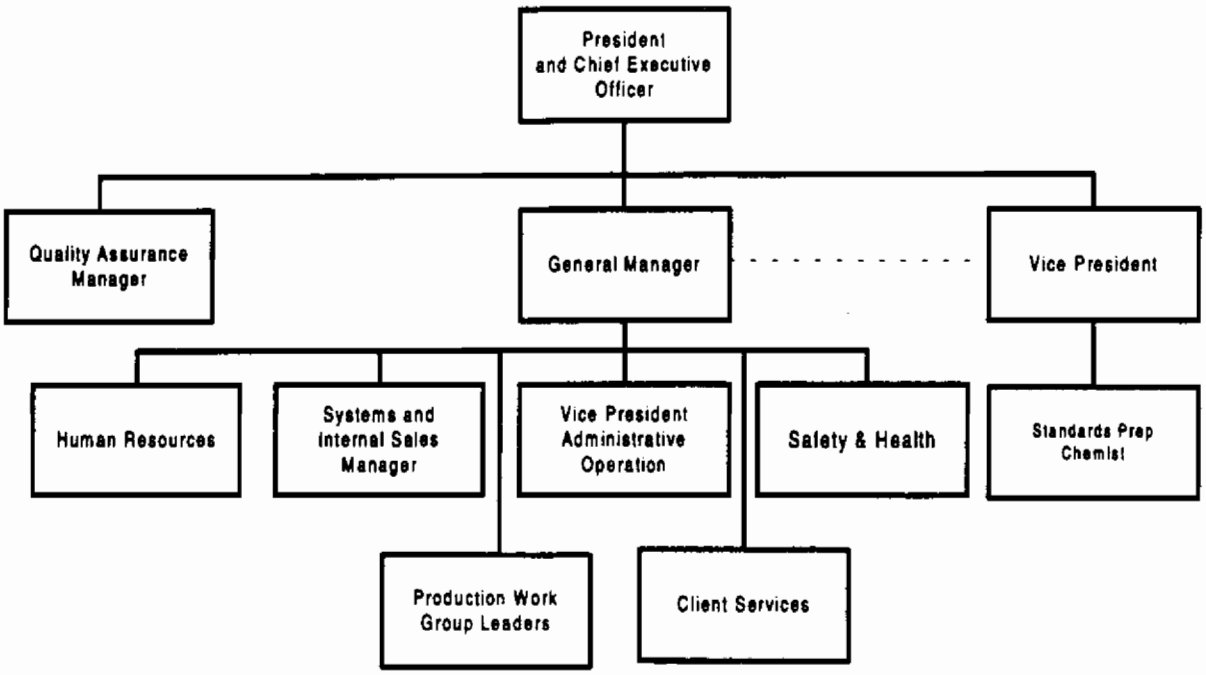
Preventive Action

Preventive actions are implemented as part of standard operating procedures, process improvement efforts and corrective actions. When circumstances inherent to a procedure are known to have a high potential for error, the SOP usually also defines measures to prevent the error from occurring. Cross-functional teams meet at Triangle Labs for the purpose of planning and implementing both process improvements and corrective actions. Information must be made available to these teams to enable them to detect, analyze and eliminate potential causes of nonconformities. Preventive actions are an integral part of corrective actions, because resultant changes in procedures often prevent recurrence of problems.

Appendix 1A

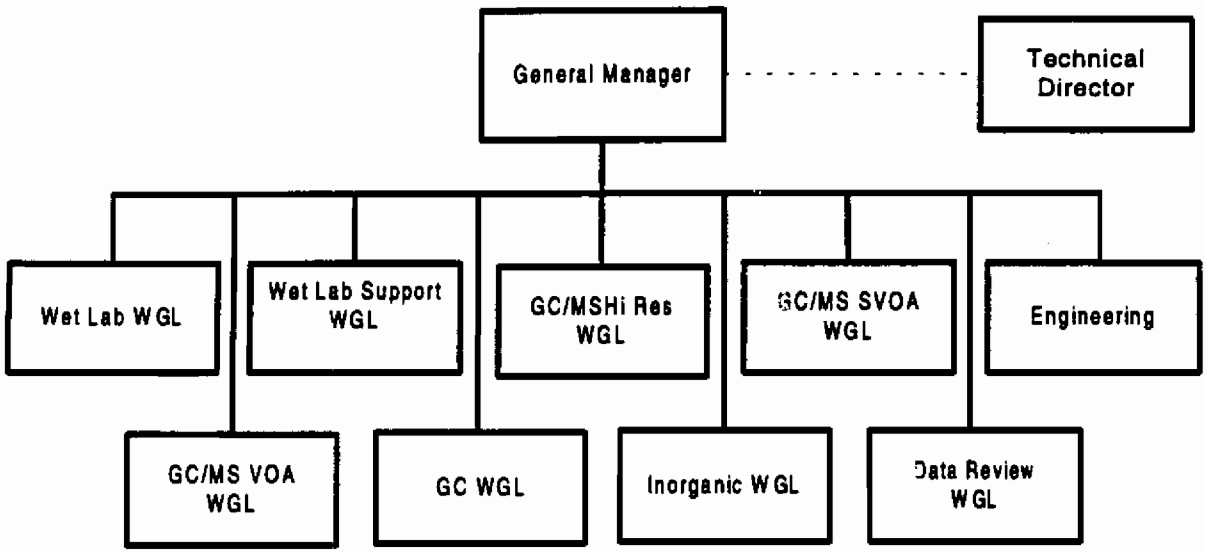
ORGANIZATIONAL CHARTS

Triangle Laboratories of RTP, Inc.



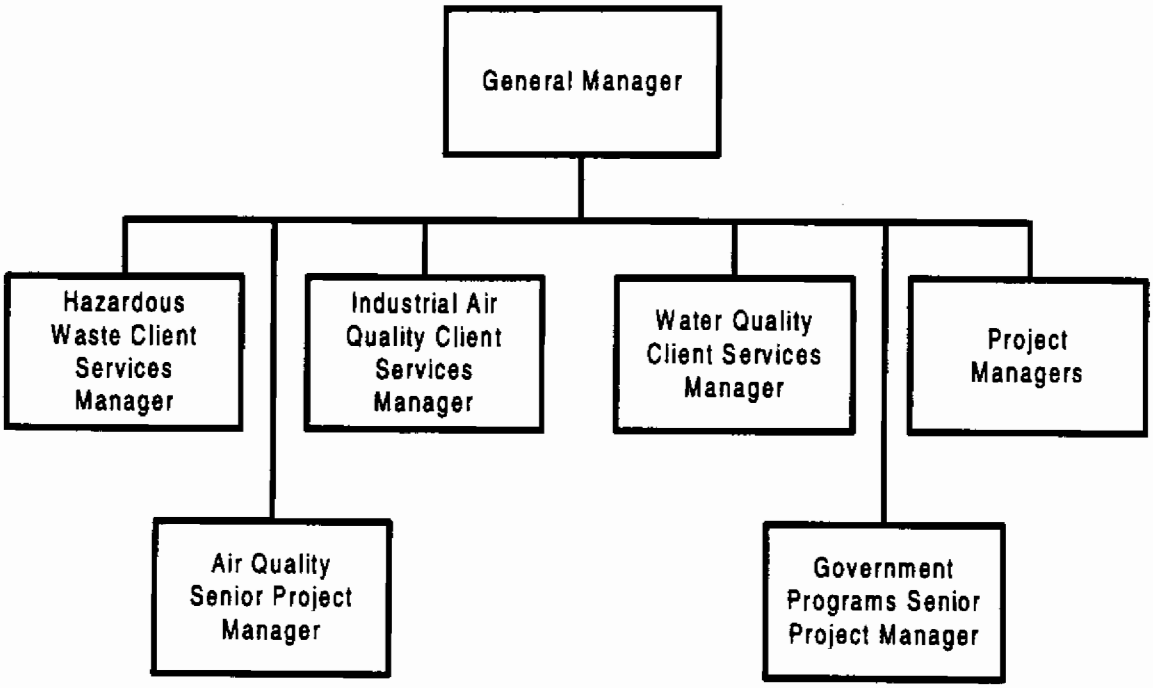
Triangle Laboratories of RTP, Inc.

Production Work Group Leaders

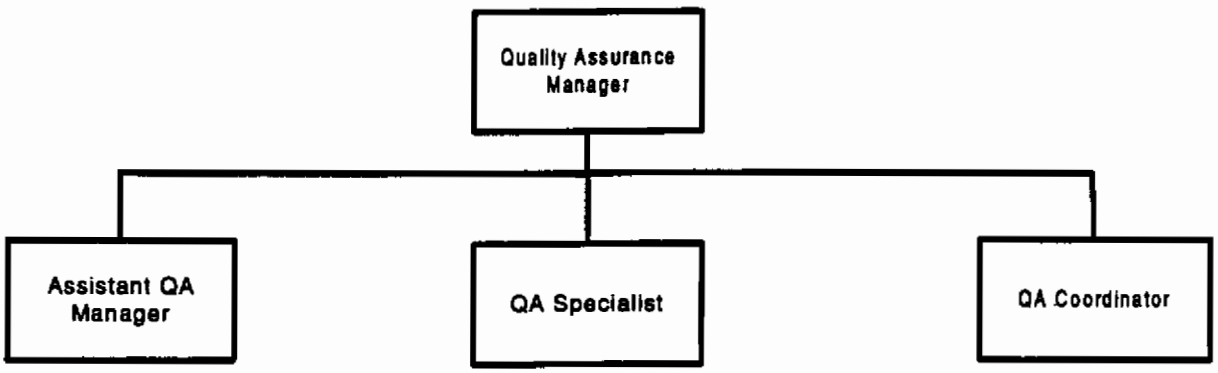


WGL = Work Group Leader

Triangle Laboratories of RTP, Inc.
Client Services

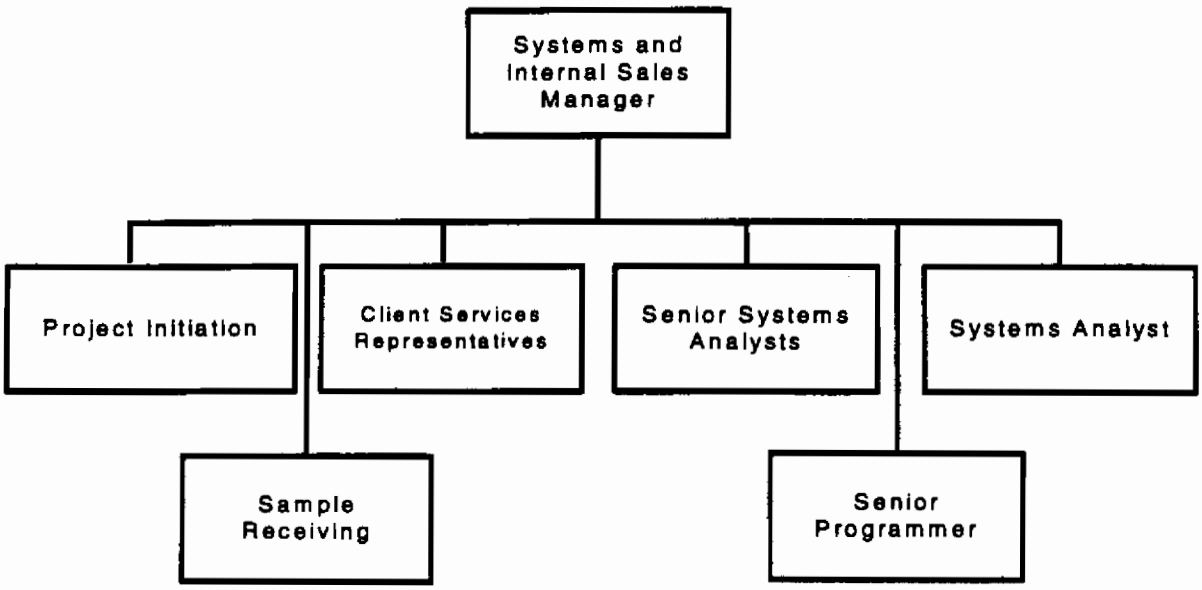


Triangle Laboratories of RTP, Inc.
Quality Assurance

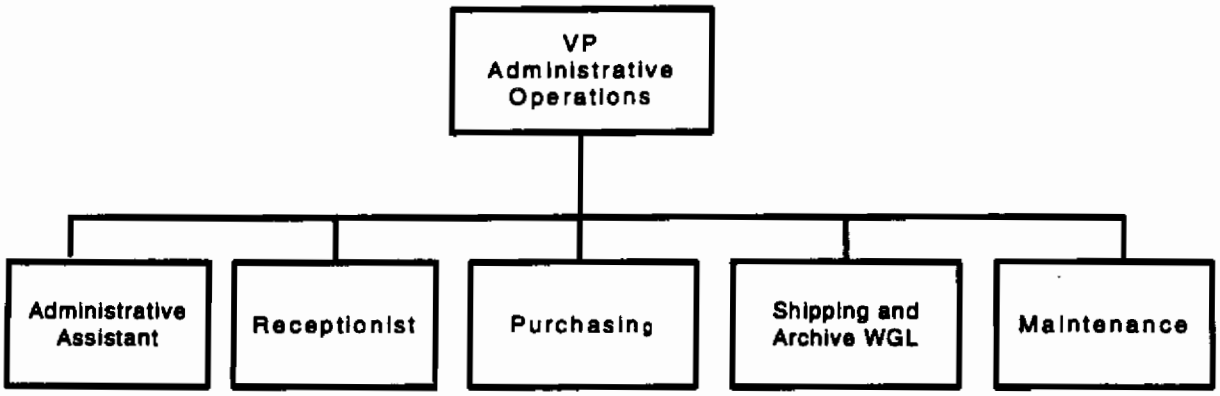


Triangle Laboratories of RTP, Inc.

Systems and Internal Sales



Triangle Laboratories of RTP, Inc.
Administrative Operations



WGL = Work Group Leader

Appendix 1B

STATE CERTIFICATIONS

State of Alaska Department of Environmental Conservation
State of Alabama Department of Environmental Management
State of Arizona Department of Health Services
State of Arkansas, Department of Pollution Control and Ecology
California Department of Health Services
State of Connecticut Department of Health Services
Delaware Health and Social Services
Florida Department of Health and Rehabilitative Services
Idaho Department of Health and Welfare
Kansas Department of Health and Environment
Commonwealth of Kentucky Department for Environmental Protection
State of Michigan Department of Public Health
Montana Department of Health and Environmental Services
New Jersey Department of Environmental Protection
State of New Mexico Environment Department
New York State Department of Health
State of North Carolina Department of Environment, Health and Natural Resources
North Dakota State Department of Health and Consolidated Laboratories
State of South Carolina Department of Health and Environmental Control
State of Tennessee Department of Environment and Conservation
Utah Department of Health
Commonwealth of Virginia Department of General Services,
Division of Consolidated Laboratory Services
State of Washington Department of Ecology
State of Wisconsin Department of Natural Resources

Appendix 2A

VOLATILE COMPOUNDS

Triangle Laboratories of RTP, Inc. analyzes for target compounds of both Methods 624 and 8240. Below are lists which indicate target compounds and surrogates common to both methods and specific to each method. Internal standard (underlined) relationships for each analyte are also indicated.

Method 624 & Method 8240, Table 2, SW-846

Bromochloromethane
Bromomethane
Chloroethane
Chloroform
Chloromethane
1,1-Dichloroethane
1,2-Dichloroethane
1,1-Dichloroethene
trans-1,2-Dichloroethene
Methylene chloride
Trichlorofluoromethane
Vinyl chloride
1,2-Dichloroethane-d_4 (surrogate)

1,4-Difluorobenzene
Benzene
Bromodichloromethane
Bromoform
Carbon tetrachloride
Chlorodibromomethane
1,2-Dichloropropane
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethene

Chlorobenzene-d_5
Chlorobenzene
Ethylbenzene
1,1,2,2-Tetrachloroethane
Tetrachloroethene
Toluene
Bromofluorobenzene (surrogate)
Toluene-d_8 (surrogate)

Method 8240, Table 2, SW-846

Bromochloromethane
Acetone
Carbon disulfide
cis-1,2-Dichloroethene
Iodomethane

1,4-Difluorobenzene
2-Butanone
Vinyl acetate
Benzene-d_6 (surrogate)[for VOST]

Chlorobenzene-d_5
2-Hexanone
4-Methyl-2-Pentanone
Styrene
o-Xylene
m-/p-Xylene

Method 624

1,4-Difluorobenzene
2-Chloroethyl vinyl ether

Chlorobenzene-d_5
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene

Non-target compounds known as tentatively identified compounds (TIC's) are identified by a computer generated search of the National Institute of Standards and Technology (NIST) Mass Spectral Library.

Appendix 2B

SEMIVOLATILE COMPOUNDS

Triangle Laboratories of RTP, Inc. analyzed for target compounds of both Methods 625 and 8270. Below is a list which indicates target compounds and surrogates common to both methods. On the following page are lists which indicate those target compounds and surrogates specific to either Method 625 or Method 8270. Internal standard (underlined) relationships for each analyte are also indicated.

Method 625 and Method 8270, Table 2, SW-846

1,4-Dichlorobenzene-d_4
Bis(2-Chloroethyl)ethene
Bis(2-Chloroisopropyl)ether
2-Chlorophenol
Hexachloroethane
N-Nitroso-di-n-propylamine
Phenol
Phenol-d_5 (surrogate)
2-Fluorophenol (surrogate)

Naphthalene-d_8
Bis(2-Chloroethoxy)methane
4-Chloro-3-methylphenol
2,4-Dichlorophenol
2,4-Dimethylphenol
Hexachlorobutadiene
Isophorone
Naphthalene
Nitrobenzene
2-Nitrophenol
1,2,4-Trichlorobenzene
Nitrobenzene-d_5 (surrogate)

Acenaphthene-d_{10}
Acenaphthene
Acenaphthylene
2-Chloronaphthalene
4-Chlorophenyl phenyl ether
Diethylphthalate
Dimethylphthalate
2,4-Dinitrophenol
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Fluorene
Hexachlorocyclopentadiene
4-Nitrophenol
2,4,6-Trichlorophenol
2-Fluorobiphenyl (surrogate)
2,4,6-Tribromophenol (surrogate)

Phenanthrene-d_{10}
Anthracene
4-Bromophenyl phenyl ether
Di-n-butylphthalate
4,6-Dinitro-2-methylphenol
Fluoranthene
Hexachlorobenzene
N-Nitrosodiphenylamine
Pentachlorophenol
Phenanthrene

Chrysene -d_{12}
Benzo(a)anthracene
Bis(2-ethylhexyl)phthalate
Chrysene
3,3'-Dichlorobenzidine
Pyrene

Perylene-d_{12}
Terphenyl-d_{14} (surrogate)
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(g,h,i)perylene
Benzo(a)pyrene
Di-n-octylphthalate
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene

Non-target compounds known as tentatively identified compounds (TIC's) are identified by a computer generated search of the National Institute of Standards and Technology (NIST) Mass Spectral Library.

Method 8270, Table 2, SW-846

1,4-Dichlorobenzene-d_4
Benzyl alcohol
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
2-Methylphenol
4-Methylphenol
1,4-Dibromobenzene-d_4 (surrogate)

Naphthalene-d_8
Benzoic acid
4-Chloroaniline
2-Methylnaphthalene
1,3,5-Trichlorobenzene-d_3 (surrogate)

Acenaphthene-d_{10}
Dibenzofuran
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
2,4,5-Trichlorophenol

Phenanthrene-d_{10}
Anthracene-d_{10} (surrogate)

Chrysene-d_{12}
Butylbenzylphthalate
Pyrene-d_{10} (surrogate)

Method 625

1,4-Dichlorobenzene-d_4
N-Nitrosodimethylamine

Chrysene-d_{12}
Benzidine

Appendix 2C

DIOXIN/FURAN COMPOUND LISTS

Table 1 - Method 551 Target Analytes

Specific Isomers	Total Isomers
2,3,7,8-TCDD	Total TCDD (22 isomers)
2,3,7,8-TCDF	Total TCDF (38 isomers)

Table 2 - Methods 8290, 23 and 1613 Target Analytes

Specific Isomers	Total Isomers
Dioxins	
2,3,7,8-TCDD	Total TCDD (22 isomers)
1,2,3,7,8-Penta-CDD	Total PeCDD (14 isomers)
1,2,3,4,7,8-Hexa-CDD	Total HxCDD (10 isomers)
1,2,3,6,7,8-Hexa-CDD	
1,2,3,7,8,9-Hexa-CDD	
1,2,3,4,6,7,8-Hepta-CDD	Total HpCDD (2 isomers)
Octa-CDD	
Furans	
2,3,7,8-TCDF	Total TCDF (38 isomers)
1,2,3,7,8-Penta-CDF	Total PeCDF (28 isomers)
2,3,4,7,8-Penta-CDF	
1,2,3,4,7,8-Hexa-CDF	Total HxCDF (16 isomers)
1,2,3,6,7,8-Hexa-CDF	
2,3,4,6,7,8-Hexa-CDF	
1,2,3,7,8,9-Hexa-CDF	
1,2,3,4,6,7,8-Hepta-CDF	Total HpCDF (4 isomers)
1,2,3,4,7,8,9-Hepta-CDF	
Octa-CDF	

Appendix 2D

PESTICIDE/PCB AND HERBICIDE COMPOUNDS

Triangle Laboratories of RTP, Inc. analyzes for target compounds of Methods 8080 and 8150. Below are lists which indicate target compounds for both methods and surrogate compounds for Method 8080.

Method 8080	Method 8150
Aldrin	2,4-D
α-BHC	2,4-DB
β-BHC	2,4,5-T
γ-BHC (Lindane)	2,4,5-TP (Silvex)
δ-BHC	Dalapon
Chlordane (technical)*	Dicamba
4,4'-DDD	Dichloroprop
4,4'-DDE	Dinoseb
4,4'-DDT	
Dieldrin	
Endosulfan I	
Endosulfan II	
Endosulfan sulfate	
Endrin	
Endrin aldehyde	
Heptachlor	
Heptachlor epoxide	
Methoxychlor	
Toxaphene	
PCB-1016	
PCB-1221	
PCB-1232	
PCB-1242	
PCB-1248	
PCB-1254	
PCB-1260	
Tetrachloro-meta-xylene (TCMX)(surrogate)	
Decachlorobiphenyl (DCBP)(surrogate)	

*Triangle Labs analyzes chlordane as specific isomers α- and γ-chlordane.

Appendix 3A

GC/MS ANALYTICAL METHODS: VOLATILES

	Method 624	Method 8240
matrices	municipal and industrial wastewater	nearly all matrices, regardless of water content
compounds	see Appendix 2A	see Appendix 2A
calibration procedures & frequency	Initial: 3 pt. minimum - if RSD <35%, use average RF, else use curve	Initial: 5 pt. minimum, SPCC compounds RF >0.300 (Bromoform >0.250), CCC compounds RSD <30% Continuing: 12 hour mid-level std., SPCC compounds RF >0.300 (Bromoform >0.250) CCC compounds %D <25% from Initial
internal standards	Bromochloromethane 1,4-Difluorobenzene Chlorobenzene-d_5	Bromochloromethane 1,4-Difluorobenzene Chlorobenzene-d_5
surrogate standards	Toluene-d_8 Bromofluorobenzene 1,2-Dichloroethane-d_4	Toluene-d_8 Bromofluorobenzene 1,2-Dichloroethane-d_4
standard solution expiration	Stock solutions: gases in MeOH - 1 week, liquids in MeOH - 1 month, dilutions in MeOH - check frequently for stability, aqueous dilutions - 24 hours with no headspace	Stock Solutions: gases in MeOH - 2 months, liquids in MeOH - 6 months, dilutions in MeOH - replace as necessary, aqueous dilutions - 1 week
holding times	within 14 days of collection	within 14 days of collection
validation	Initial Performance Analyses (water): Four 5 ml aliquots of QC Check Sample composed of reagent water spiked with all analytes at 20 µg/L, results must meet acceptance criteria for the method	
QC check sample	One sample aliquot, spiked with all analytes at 20 µg/L, results must meet acceptance criteria for the method. Frequency: every 20 samples or once per month, whichever is greater; For Method 624, the QC check sample is used for verification of the initial calibration	
matrix spike analysis	One sample aliquot spiked at 20 µg/L or 1 to 5 times the predetermined background analyses concentration in the sample, results must meet acceptance criteria for the method. Frequency: every 20 samples or once per month, whichever is greater	

Appendix 3B

GC/MS ANALYTICAL METHODS: SEMIVOLATILES

	Method 625	Method 8270
matrices	municipal and industrial wastewater	solid waste matrices, soils, groundwater
compounds	see Appendix 2B	see Appendix 2B
calibration procedures & frequency	Initial: 3 pt. minimum - if RSD <35%, use average RF, else use curve Continuing: 12 hour mid-level standard must be ≤20% D from Initial	Initial: 5 pt. minimum, SPCC compounds RF ≥0.050, CCC compounds RSD <30% Continuing: 12 hour mid-level standard, SPCC compounds RF ≥0.050, CCC compounds %D <30% from Initial
internal standards	1,4-Dichlorobenzene-d_4, Naphthalene-d_8, Acenaphthene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12}, Perylene-d_{12}	
surrogate standards	Nitrobenzene-d_5 2-Fluorobiphenyl Terphenyl-d_{14} Phenol-d_5 2-Fluorophenol 2,4,6-Tribromophenol	same as Method 625, plus: Pyrene-d_{10} 1,3,5-Trichlorobenzene-d_3 1,4-Dibromobenzene-d_4 Anthracene-d_{10}
standard solution expiration	Stock standard solutions must be replaced after six months, or sooner if comparison to QC check samples indicates a problem	Stock standard solutions must be replaced after one year, or sooner if comparison to QC check samples indicates a problem
holding times	Extraction: within 7 days of collection for waters, within 14 days of collection for soils; Analysis: within 40 days of extraction	
validation	Initial Performance Analyses (water): Replicate set of four 1 liter QC check samples composed of reagent water spiked with all analytes at 100 µg/L, extracted and analyzed. Results must meet acceptance criteria for the method	
QC check sample	A one liter QC check sample, composed of reagent water spiked with all analytes at 100 µg/L, extracted and analyzed. Results must meet acceptance criteria for the method. Frequency: every 20 samples or once per month, whichever is greater	
matrix spike analyses	One sample aliquot spiked at 100 µg/L or 1 to 5 times the predetermined background concentration in the sample. Results must meet acceptance criteria for the method. Frequency: every 20 samples or once per month, whichever is greater	

Appendix 3C

GC/MS ANALYTICAL METHODS: DIOXIN/FURAN

	Method 8290/23	Method 1613	Method 551
matrices	water, soil, sludge, tissue, pulp, paper, ash, MM5, PUF (MM5 only for Method 23)		
compounds	see Appendix 2C, Table 2	see Appendix 2C, Table 2	see Appendix 2C, Table 1
calibration procedures & frequency	Initial: 5 points, 20/25% RSD (25/30% RSD for Method 23) Continuing: mid-level standard every 12 hours, 20/25% RPD (25/30% RPD for Method 23)	Initial: 5 points, 20/25% RSD Continuing: mid-level standard every 12 hours, 20/25% RPD	Initial: 5 points in duplicate, 20/25% RSD Continuing: mid-level standard every 12 hours and 4th point standard at end of injection sequence, 20/25% RPD
internal standards	$^{13}C_{12}$-2,3,7,8-TCDF $^{13}C_{12}$-2,3,7,8-TCDD $^{13}C_{12}$-1,2,3,7,8-PeCDF $^{13}C_{12}$-1,2,3,7,8-PeCDD $^{13}C_{12}$-1,2,3,6,7,8-HxCDF $^{13}C_{12}$-1,2,3,6,7,8-HxCDD $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD $^{13}C_{12}$-1,2,3,4,6,7,8,9-OCDD	same as Method 8290, plus: $^{13}C_{12}$-2,3,4,7,8-PeCDF $^{13}C_{12}$-1,2,3,4,7,8-HxCDF $^{13}C_{12}$-1,2,3,7,8,9-HxCDF $^{13}C_{12}$-2,3,4,6,7,8-HxCDF $^{13}C_{12}$-1,2,3,4,7,8-HxCDD $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	
surrogate standards	$^{13}C_{12}$-2,3,4,7,8-PeCDF $^{13}C_{12}$-1,2,3,4,7,8-HxCDF $^{13}C_{12}$-1,2,3,4,7,8-HxCDD $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF $^{37}Cl_4$-2,3,7,8-TCDD		
standard solution expiration	Neat standards: expiration time of 5 years, or according to supplier specification Stock standards: concentrations ≥10 ng/µL must be replaced after 2 years, concentrations ≥0.1 to <10 ng/µL must be replaced after 1 year Working standards: concentrations ≥0.1 to <1 ng/µL must be replaced after 6 months, concentrations <0.1 ng/µL must be replaced after 3 days		
holding times	Extraction within 7 days of collection, analysis within 40 days of extraction		
validation	For Method 1613 waters only: Initial Precision and Recovery: replicates of four 1 liter QC check samples fortified with all specific isomers: tetra at 200 pg/L, penta through hepta at 1,000 pg/L, and octa at 2,000 pg/L For Method 23 only: an EPA audit sample submitted by the client with each sample batch or alternatively, prepared in-house from EPA supplied ampules		
QC check sample	For Method 1613 waters only: Ongoing Precision and Recovery: one QC check sample of 1 liter fortified with all specific isomers: tetra at 200 pg/L, penta through hepta at 1000 pg/L, and octa at 2000 pg/L; Frequency: once per batch of 20 samples or less		
MS/MSD analysis	Accuracy: ±30%, Precision: high level - RPD = ±25%, low level - RPD = ±50%		

Appendix 3D

GC ANALYTICAL METHODS:PESTICIDES/PCBs AND HERBICIDES

	Method 8080	Method 8150
matrices	groundwater, soil, non water-miscible waste	groundwater, soil, sediment, other solids and wastes
compounds	see Appendix 2D	see Appendix 2D
calibration procedures & frequency	Initial: 5 point minimum, RSD ≤20%, use average RF Continuing: mid-level standard every 10 samples, %D ≤15%	Initial: 5 point minimum RSD ≤20%, use average RF Continuing: mid-level standard every 10 samples, %D ≤15%
surrogate standards	Decachlorobiphenyl 2,4,5,6-Tetrachloro-meta-xylene	One or two herbicide surrogates not expected to be present in samples
holding times	Extraction: within 7 days of collection for waters; within 14 days of collection for soils Analysis: within 40 days of extraction	Extraction: within 7 days of collection for waters; within 14 days of collection for soils Analysis: within 40 days of extraction
validation	Initial Performance Analyses (water): Replicate set of four 1 liter QC Check Samples, composed of reagent water spiked with all analytes at 10 and 2 ug/L, extracted and analyzed; results must meet acceptance criteria for the method	Initial Performance Analyses (water): Replicate set of four 1 liter QC Check Samples, composed of reagent water spiked with all analytes at 10 and 2000 ug/L, extracted and analyzed; results must meet acceptance criteria for the method
QC check sample	A 1 liter QC check sample, composed of reagent water spiked with all analytes at 10 and 2 µg/L, extracted and analyzed; Results must meet acceptance criteria for the method; Frequency: every 10 samples or once per month, whichever is greater	A one liter QC Check sample, composed of reagent water spiked with all analytes at 10 and 2000 µg/L, extracted and analyzed; Results must meet acceptance criteria for the method; Frequency: every 10 samples or once per month, whichever is greater
matrix spike analyses	One sample aliquot spiked at 10 and 2 µg/L or 1 to 5 times the predetermined background concentration in the sample; Results must meet acceptance criteria for the method; Frequency: every 10 samples or once per month, whichever is greater	One sample aliquot spiked at 10 and 2000 µg/L or 1 to 5 times the predetermined background concentration in the sample; Results must meet acceptance criteria for the method; Frequency: every 10 samples or once per month, whichever is greater

Appendix 4A

STANDARD OPERATING PROCEDURE CATEGORIES

ACC - Accounting
ADO - Administrative Operations
AGN - Administrative General
AQU - Air Quality Preparation
BMD - Business Management Department
CGN - Corporate General
CMA - Corporate Maintenance
CQA - Corporate Quality Assurance
CSS - Computer System Support
DDR - Dioxin Data Review
DHC - HRGC/HRMS PCBs
DHP - HRGC/HRMS PAHs
DHR - Dioxin HRGC/HRMS
DMD - Dioxin Management Department
DPA - Data Package Assembly
DRG - Dioxin Report Generation
DSP - Dioxin Sample Preparation
IIC - Ion Chromatography
INO - Inorganics
OAO - Organics AOX/TOX
ODS - Organic and Dioxin Standards
OGC - Organics GC
OMS - Organics GC/MS
ORG - Organics Report Generation
OWL - Organics Wet Lab
PPC - Production Process Control
PSH - Personnel, Safety and Health
SCS - Sales and Client Services
SMC - Sample Custodian
TGN - Technical General

Appendix 5A

Containers, Preservatives and Holding Times

Parameter	Matrix	Holding time	Container	Preservative[d]
Volatile[a] organics	Water	14 days	Two 40 mL glass vials Teflon lined septum	4 drops conc.HCl 4°C
	Soil	14 days	Brass or Teflon core tube, sealed both ends	4°C
Extractable[b] organics	Water	7 days until extraction; 40 days after extraction	1 L glass with Teflon liner	4°C
	Soil	14 days until extraction; 40 days after extraction	Glass jar with Teflon liner or core tube	4°C
Metals[c] (other than mercury)	Water	6 months	polyethylene or glass	HNO_3 to pH <2
	Soil	6 months	polyethylene or glass	4°C
Mercury	Water	28 days	polyethylene or glass	HNO_3 to pH <2
	Soil	28 days	polyethylene or glass	4°C

[a]For SW-846: Free chlorine must be removed prior to addition of HCl by exact addition of $Na_2S_2O_3$. Adjust pH <2 for purgeable aromatic hydrocarbons with H_2SO_4, HCl or solid $NaHSO_4$. Adjust pH to 4-5 for acrolein and acrylonitrile.

[b]For SW-846: Preserve phenols, benzidines, nitrosamines, nitroaromatics and cyclic ketones, PAHs, haloethers, chlorinated hydrocarbons and pesticides with 0.008% $Na_2S_2O_3$. Nitrosamines, Nitroaromatics, cyclic ketones and PAHs should be stored in the dark. Pesticides pH=5-9.

[c]For CLP: Dissolved metals require filtration before pH adjustment.

[d]Preservation temperatures are approximate with an acceptable range of ±2°C.

Appendix 6A

LABORATORY NOTEBOOKS

The following is a list of many laboratory notebooks maintained at Triangle Laboratories. Each logbook is maintained according to the general SOP No. CGN104.

Area	Notebook(s) Maintained:	Area:	Notebook(s) Maintained:
Receiving	Sample Receipt	High Res Data Review	ConCal ICAL
Low Res Wet Laboratory	Gel Permeation Chromatography Balance and Pipette Gross Sample Weight Florisil Standardization pH Solution Preparation Extract Archive	Gas Chromatography Lab	Instrument Run Log Maintenance Log
		High Res Wet Laboratory	Balance Calibration Sample Weight Solution Preparation Packing Materials Spike/Dilution Sample Transfer Sample Archive Sample Import Sample Re-extract
Low Res GC/MS	Instrument Run Log Maintenance Log Balance Log Standards Standards Receipt		
High Res GC/MS	Instrument Run Log Maintenance Log	Sample Container Preparation	XAD/PUF Blanks XAD/PUF/Filter Shipping and Inventory VOST Shipment Thermal Conditioning Sampling Kit Shipment
AOX/TOX	Instrument Run Log Maintenance Log Standards		
Standard Preparation	Standards	Quality Assurance Unit	GLP Critical Phase Inspection Activity CAR Control
Low Res Data Review	Report Preparation	Shipping and Archive	Sample Shipping Archive Storage Log Secured Area Entrance

Appendix 7A

TRIANGLE LABORATORIES OF RTP, INC.
CORRECTIVE ACTION REPORT

CAR No.:_____ Originator:_____ Date:_____

Recipient:_____

Problem Identification: Describe the problem clearly, making attachments as necessary. Send the CAR to the recipient and a copy to the QAU.

Cause Investigation: Describe the apparent cause of the problem.

Corrective/Preventive Action: Describe actions planned or completed, and follow-up plans, including a schedule for completion. Make attachments as necessary.

C.A. Analyst:_____ Date:_____

Approval:
Manager:_____ Date:_____

Follow-up:
C.A. Analyst:_____ Date:_____

QA Manager:_____ Date:_____